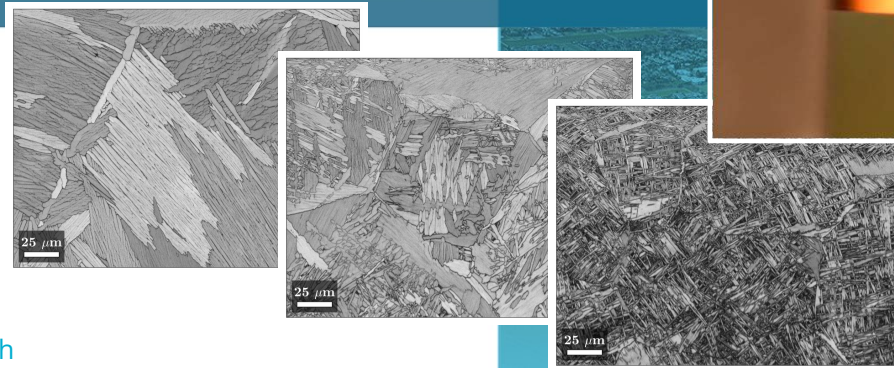
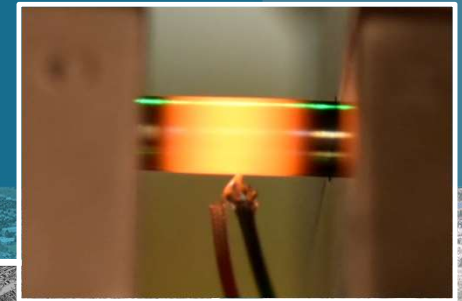




# Gleeble Dilatometry to Resolve Microstructure Evolution in W-DED Ti-6Al-4V



**Chris Finfrock, [cfinfro@sandia.gov](mailto:cfinfro@sandia.gov)**

Jessica Buckner, Jonathan Pegues, Hannah Sims, Jack Herrmann, Anthony Marcon

2024 TMS Annual Meeting & Exhibition  
 Additive Manufacturing: Process-induced Microstructures and Defects  
 March 6, 2024



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## SNL is considering WAAM and EBAM Ti-6Al-4V for structural components

### Recall: TMS2022

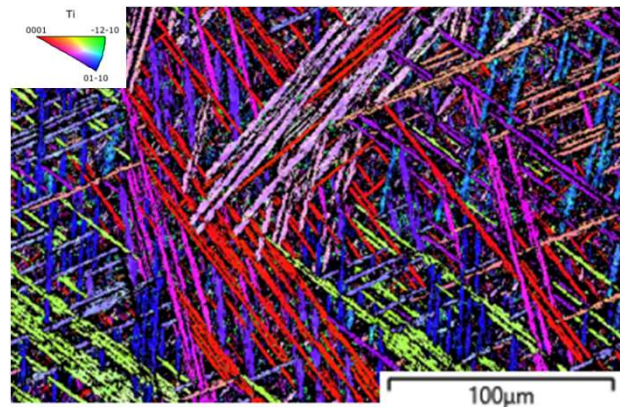
"Microstructure plays a greater role in failure than defects for both W-DED processes"

#### Heat Treatment Effects on Mechanical Properties of Wire Arc Additive Manufactured and Electron Beam Additive Manufactured Ti-6Al-4V

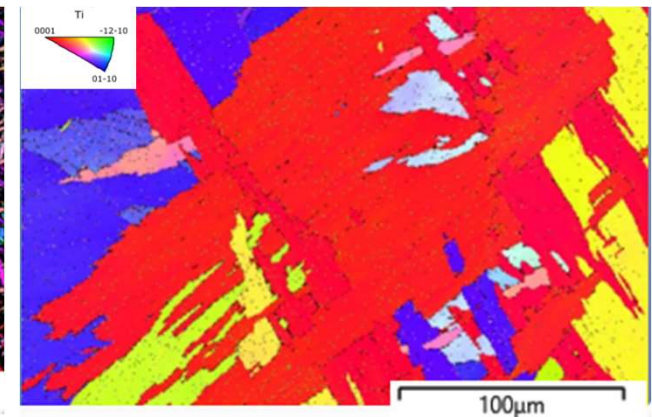
Jonathan Pegues, Shaun Whetten,  
Andrew B. Kustas, William Dannemann,  
Tyler Chilson

 Sandia National Laboratories

Electron beam additive manufactured (EBAM)



Wire arc additive manufactured (WAAM)



Notionally identical  $\beta$ -annealing processes generated unique microstructures and properties!

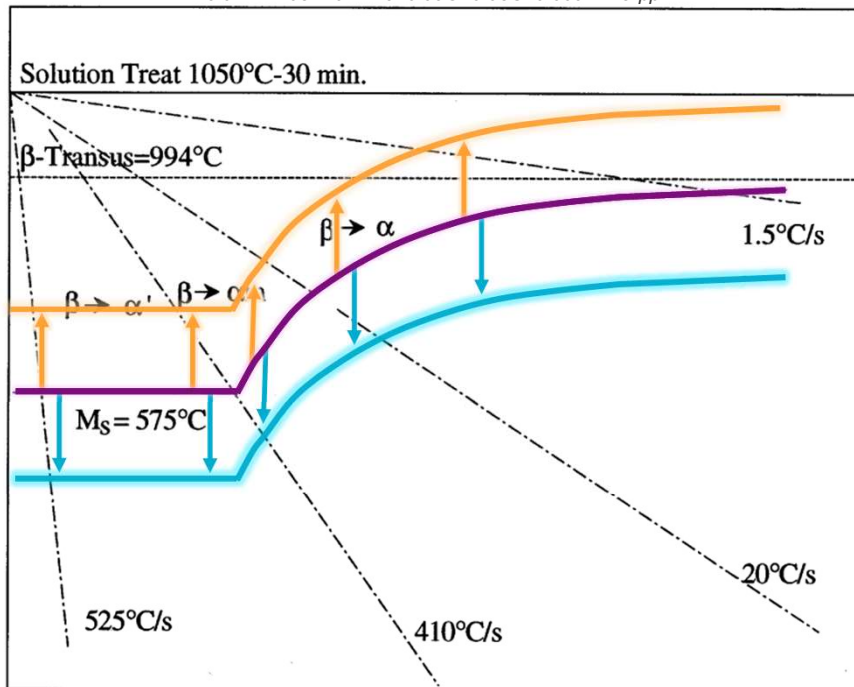
Deepening our understanding of microstructural evolution during heat treatment will facilitate thermal processing enhancements that improve performance in service.



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## Multiple competing phase transformations are operative for cooling rates relevant to thermal processing of AM Ti-6Al-4V

T. Ahmed and H.J. Rack, MSEA, 1998  
*Instrumented Jominy end quench bar*  
 Ti-6.04Al-4.03V-0.12Fe-0.09O-0.03C-0.009N-23 ppm H



G. Lütjering and J. Williams, 2007

The temperature of the  $\beta \rightarrow \alpha/\alpha'$  phase transformation exists around 850°C under quasi-equilibrium cooling rates. It increases with increasing amounts of  $\alpha$  stabilizers and decreases with increasing  $\beta$  stabilizer content"

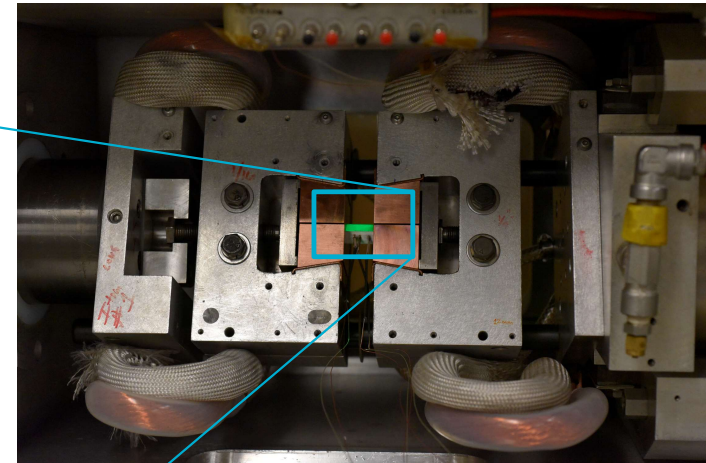
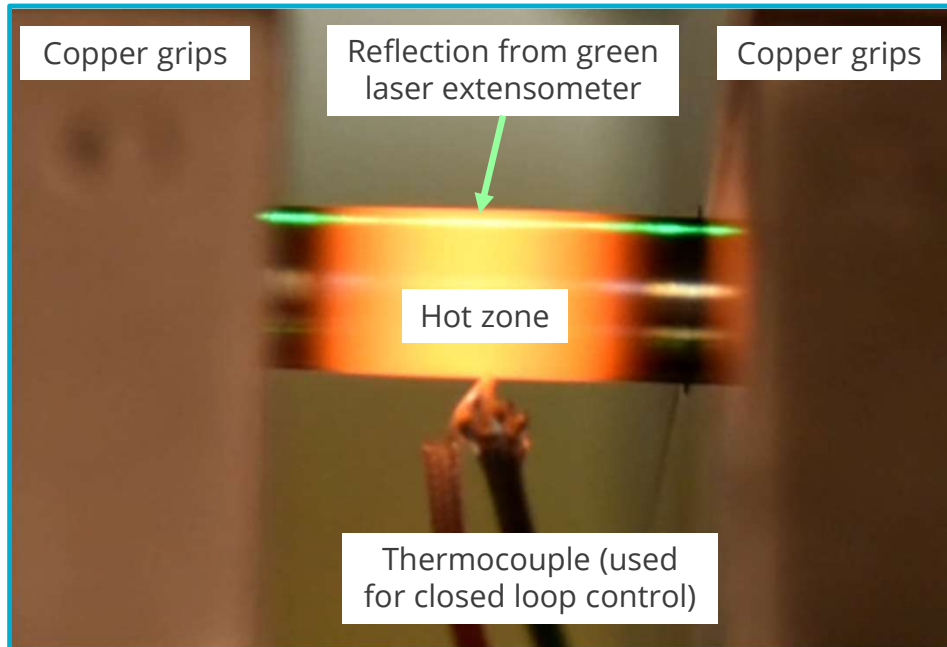
$\beta$  stabilizers:

$$[\text{Mo}]_{\text{eq.}} = [\text{Mo}] + 0.2[\text{Ta}] + 0.28[\text{Nb}] + 0.4[\text{W}] + 0.67[\text{V}] + 1.25[\text{Cr}] + 1.25[\text{Ni}] + 1.7[\text{Mn}] + 1.7[\text{Co}] + 2.5[\text{Fe}].$$

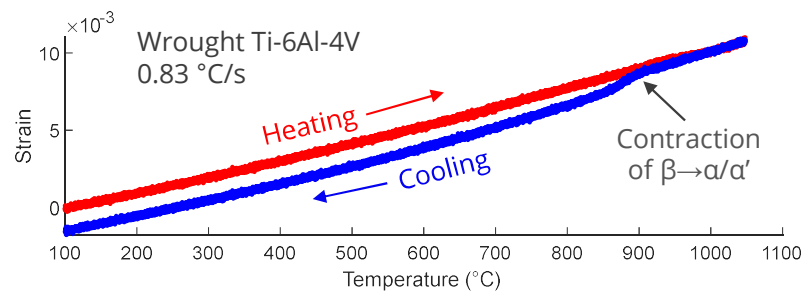
$\alpha$  stabilizers:

$$[\text{Al}]_{\text{eq.}} = [\text{Al}] + 0.17[\text{Zr}] + 0.33[\text{Sn}] + 10[\text{O}]$$

## Quench dilatometry in the Gleeble



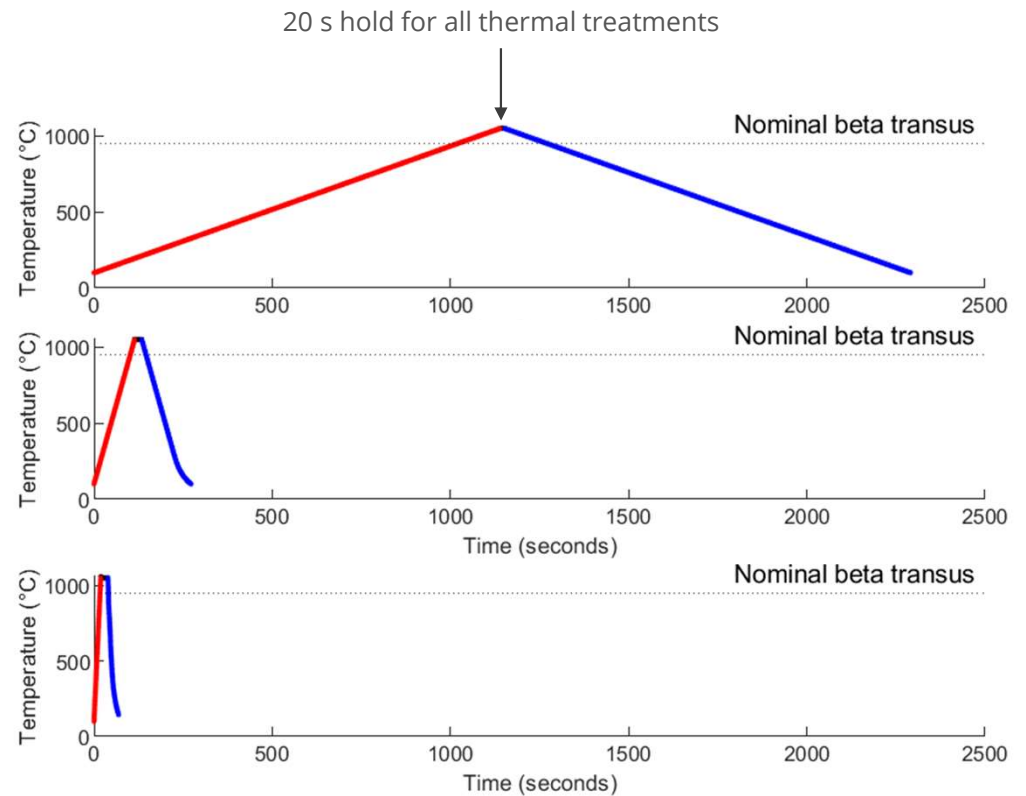
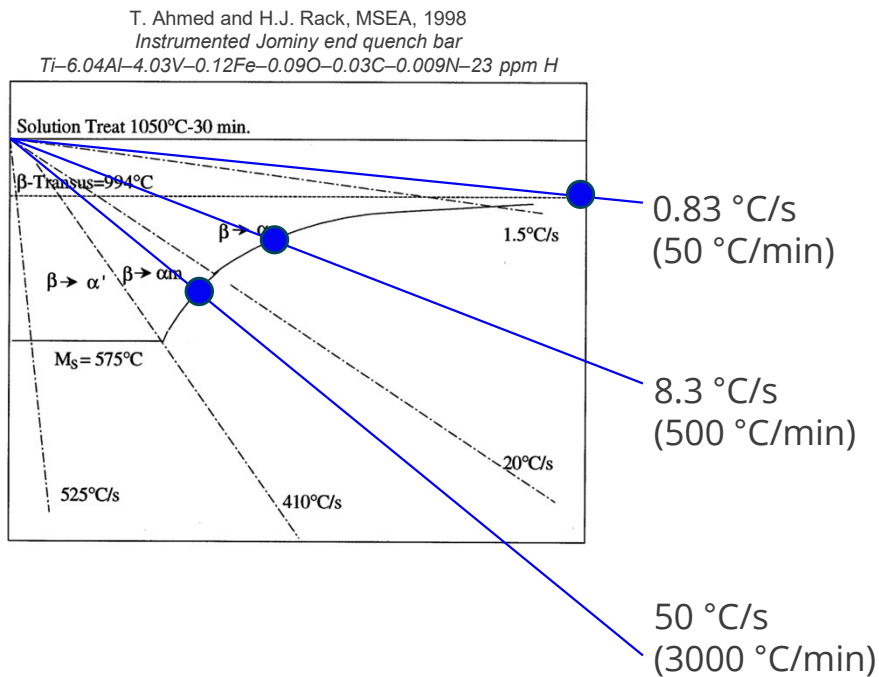
- The Gleeble 3500 is a commercially-available thermo-physical simulator that can rapidly modify specimen temperature and strain state. It was designed to simulate forging and welding processes.
- In this experiment, we rapidly heated specimens using joule heating in a high-cleanliness vacuum. A non-contact laser extensometer was used to measure volume changes that could be associated with phase transformations. High cooling rates were accomplished using an argon gas quench.
- No external force was applied, although in other experimental configurations, we can push and pull samples like in a conventional servo-hydraulic load frame.





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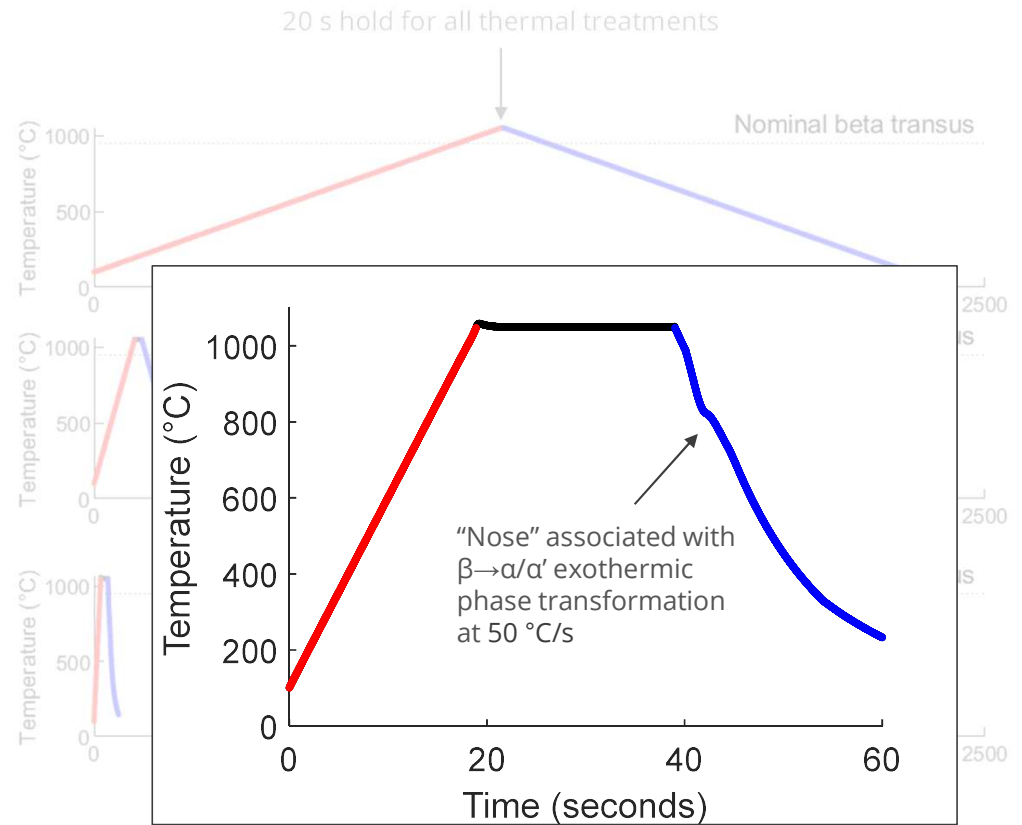
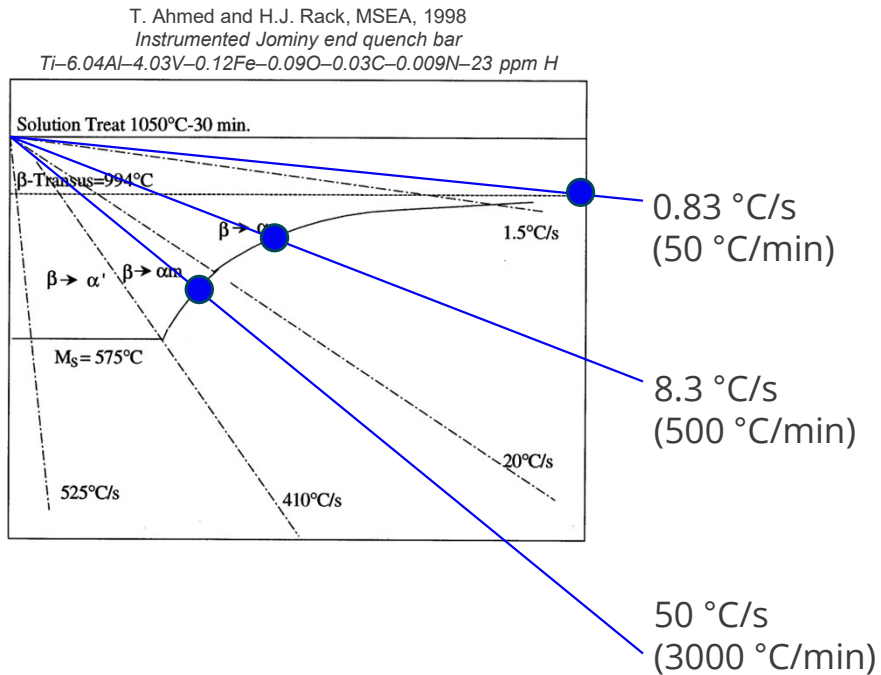
## We simulated cooling rates that span a wide range of heat treatment possibilities





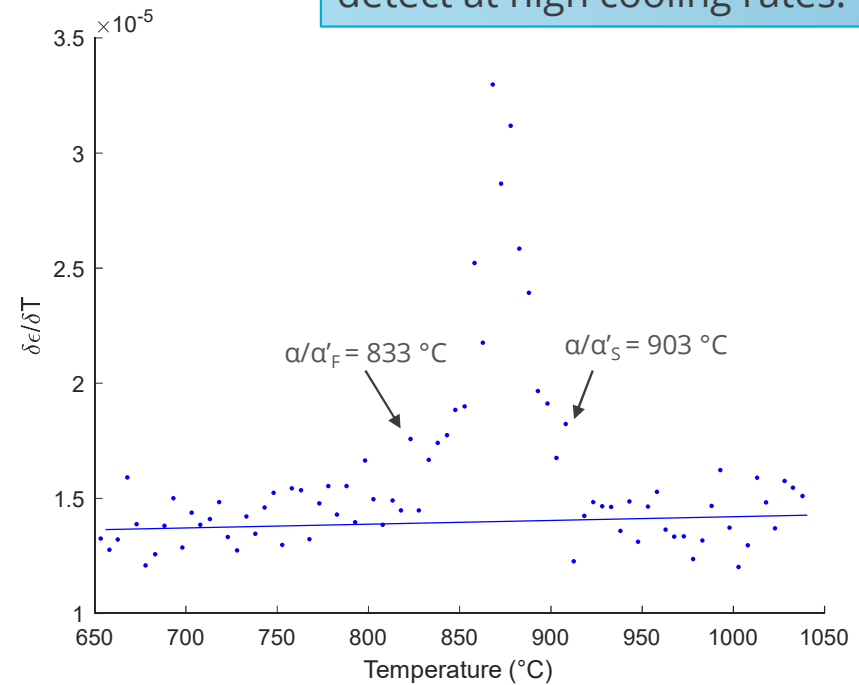
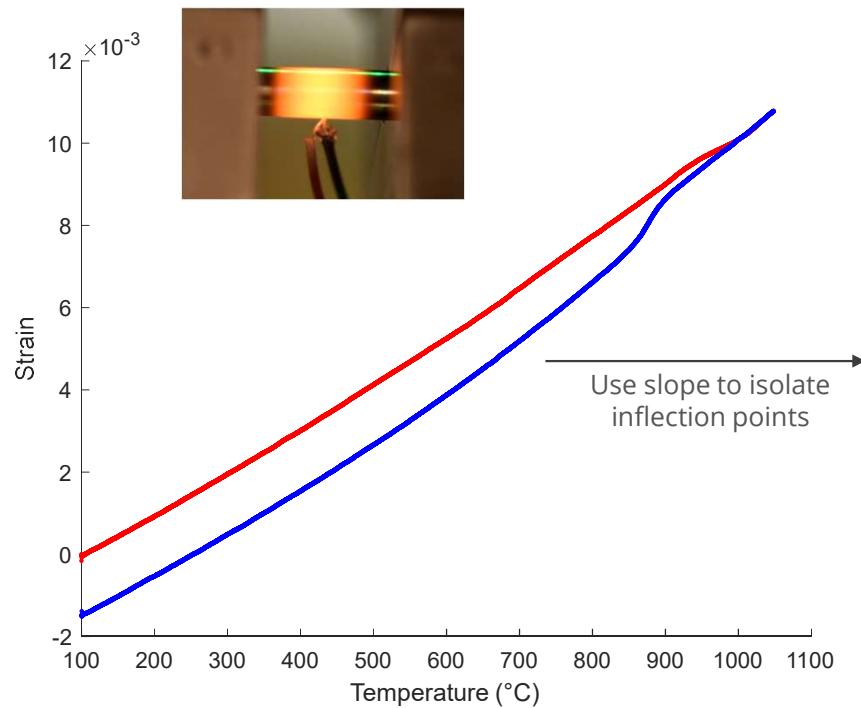
6

## We simulated cooling rates that span a wide range of heat treatment possibilities





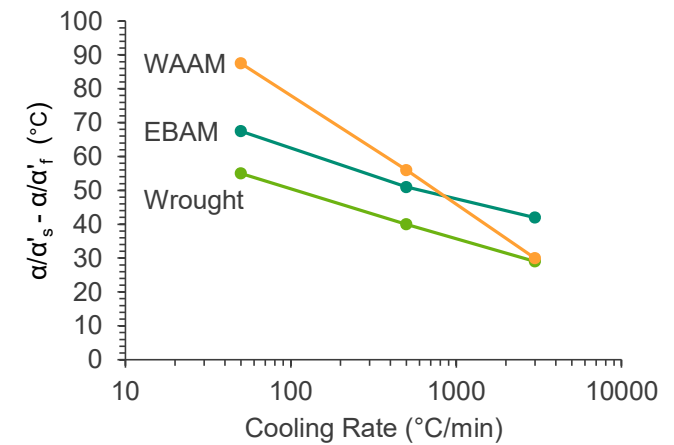
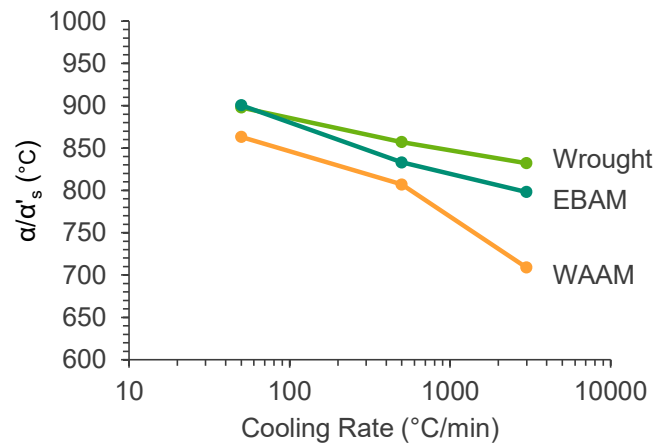
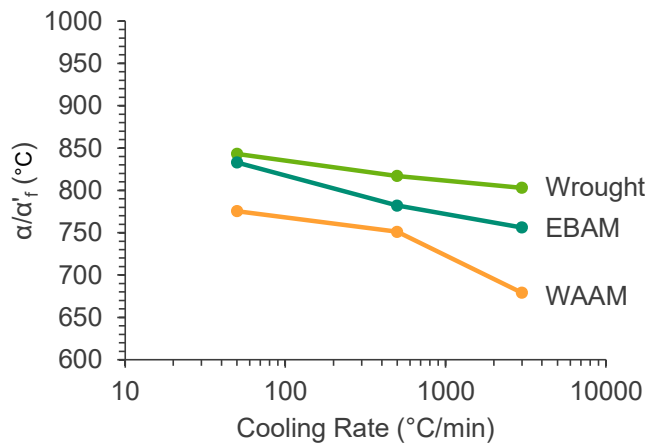
## 7 $\beta \rightarrow \alpha/\alpha'$ transformation temperatures responded to simulated cooling rates and AM processes





8

## $\beta \rightarrow \alpha/\alpha'$ transformation temperatures responded to simulated cooling rates and AM processes

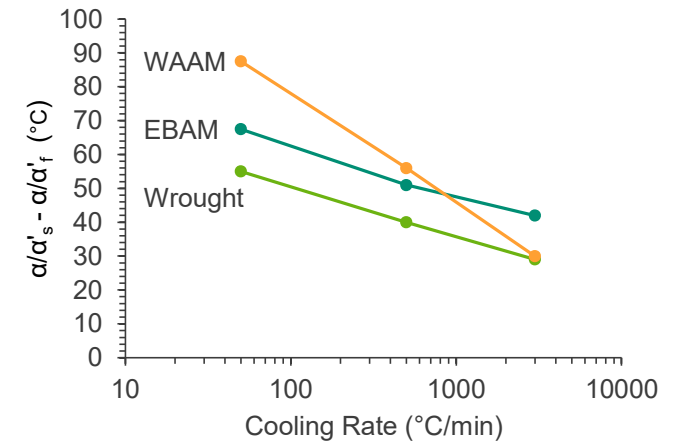
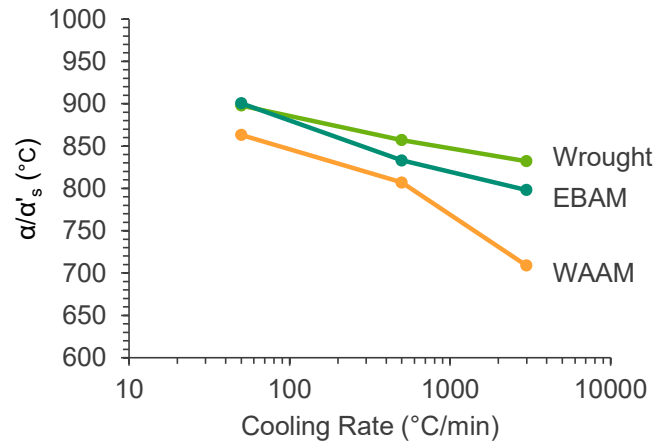
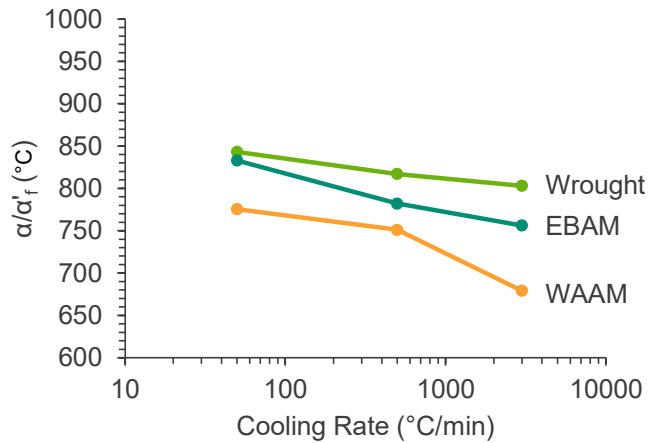


Material	Alpha Transus °C	Beta Transus °C
Wrought	853	1033
EBAM	815	1013
WAAM	823	982

\*Measured on heating near equilibrium with TGA.



## 9 $\beta \rightarrow \alpha/\alpha'$ transformation temperatures responded to simulated cooling rates and AM processes



Material	Alpha Transus °C	Beta Transus °C	Al wt. %	V wt. %	Fe wt. %	C wt. %	N wt. %	O wt. %	H wt. %	[Mo]eq.	[Al]eq.
Wrought	853	1033	6.48	4.02	0.18	0.021	0.0076	0.17	0.0036	3.14	8.18
EBAM	815	1013	6.22	4.02	0.18	0.25	0.0093	0.17	0.0011	3.14	7.92
WAAM	823	982	6.21	4.14	0.12	0.013	0.011	0.072	0.0019	3.07	6.93

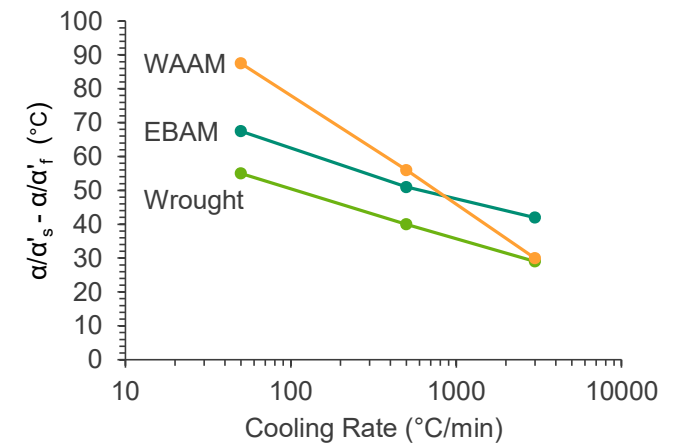
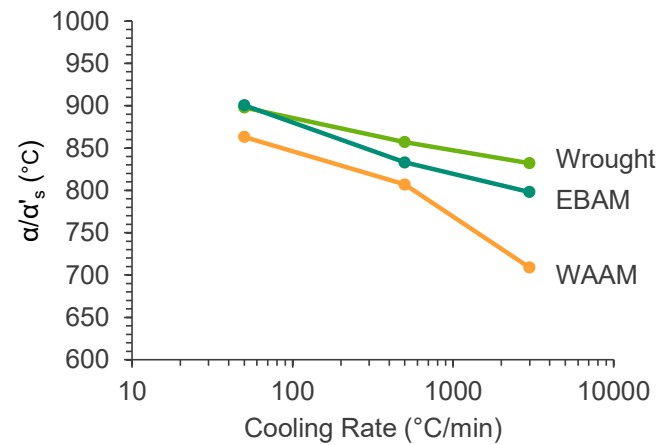
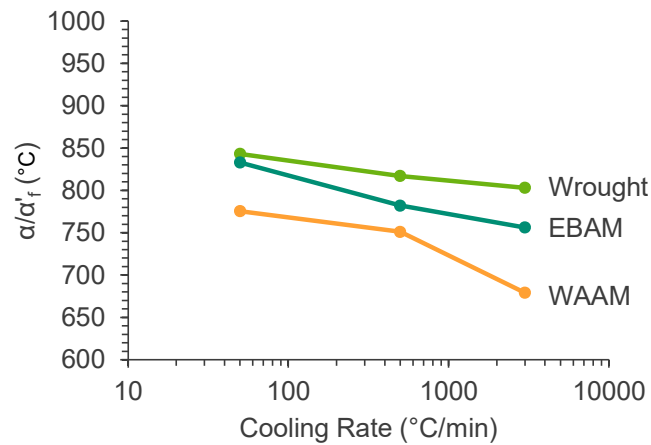
$\beta$  stabilizers:  $[Mo]eq. = 0.67 [V] + 2.5 [Fe]$ .

*Adapted after Lütjering and Williams*



10

## $\beta \rightarrow \alpha/\alpha'$ transformation temperatures responded to simulated cooling rates and AM processes



Material	Alpha Transus °C	Beta Transus °C	Al wt. %	V wt. %	Fe wt. %	C wt. %	N wt. %	O wt. %	H wt. %	[Mo]eq.	[Al]eq.
Wrought	853	1033	6.48	4.02	0.18	0.021	0.0076	0.17	0.0036	3.14	8.18
EBAM	815	1013	6.22	4.02	0.18	0.25	0.0093	0.17	0.0011	3.14	7.92
WAAM	823	982	6.21	4.14	0.12	0.013	0.011	0.072	0.0019	3.07	6.93

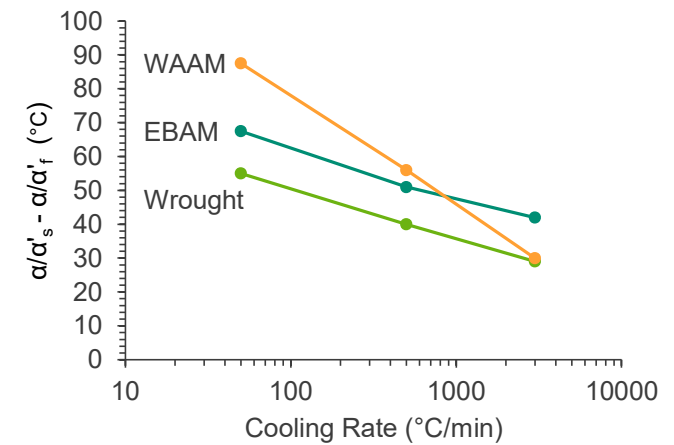
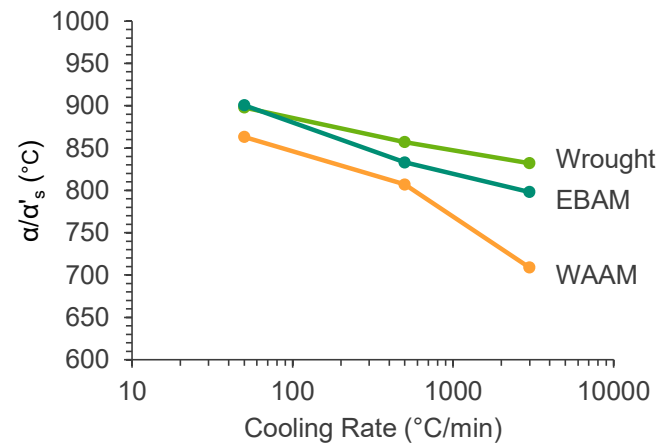
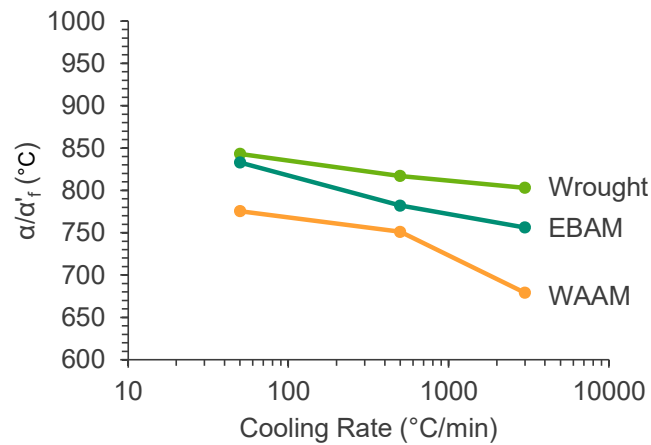
$\alpha$  stabilizers:  $[Al]_{eq.} = [Al] + 10 [O]$

*Adapted after Rosenberg*



11

## $\beta \rightarrow \alpha/\alpha'$ transformation temperatures responded to simulated cooling rates and AM processes



Material	Alpha Transus °C	Beta Transus °C	Al wt. %	V wt. %	Fe wt. %	C wt. %	N wt. %	O wt. %	H wt. %	[Mo]eq.	[Al]eq.
Wrought	853	1033	6.48	4.02	0.18	0.021	0.0076	0.17	0.0036	3.14	8.18
EBAM	815	1013	6.22	4.02	0.18	0.25	0.0093	0.17	0.0011	3.14 (0%)	7.92 (-3.2%)
WAAM	823	982	6.21	4.14	0.12	0.013	0.011	0.072	0.0019	3.07 (-2.2%)	6.93 (-15.3%)



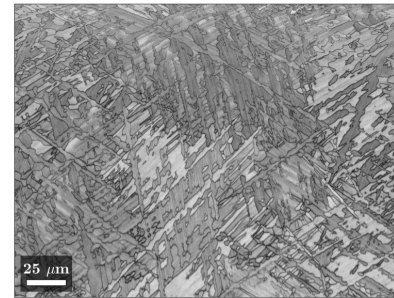
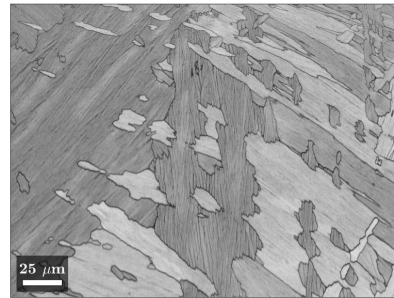
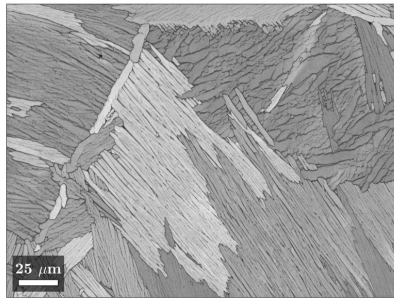
12

# Microstructure was sensitive to both cooling rate and processes

Wrought

EBAM

WAAM



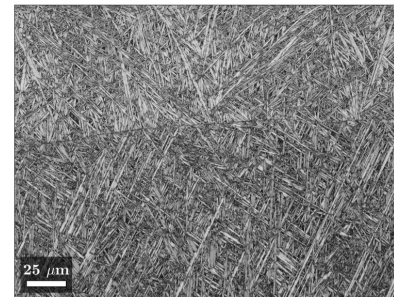
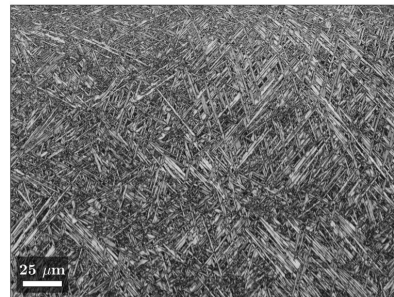
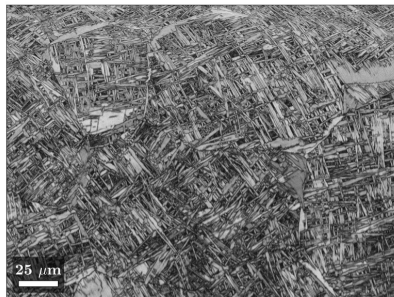
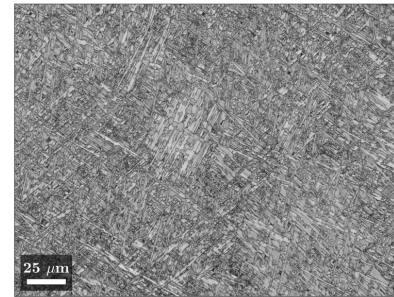
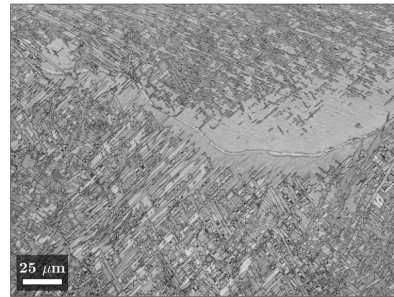
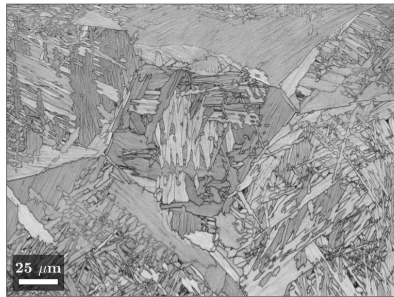
Build Direction →

**$\beta$  transformation products at increasing cooling rates:**

Widmanstätten  $\alpha+\beta$

↓  
Massive  $\alpha'$

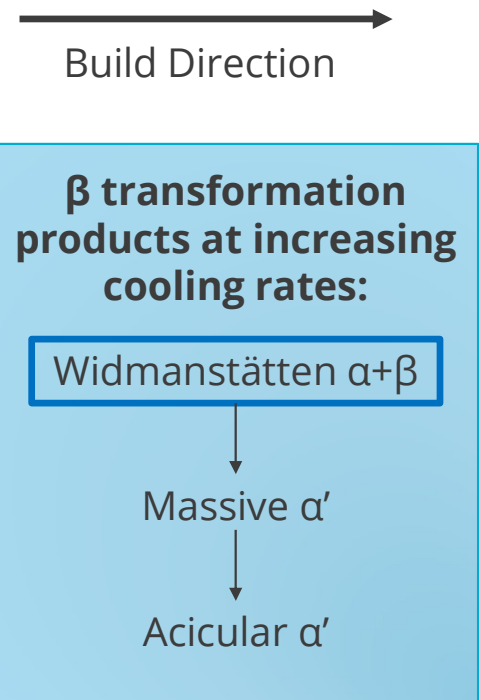
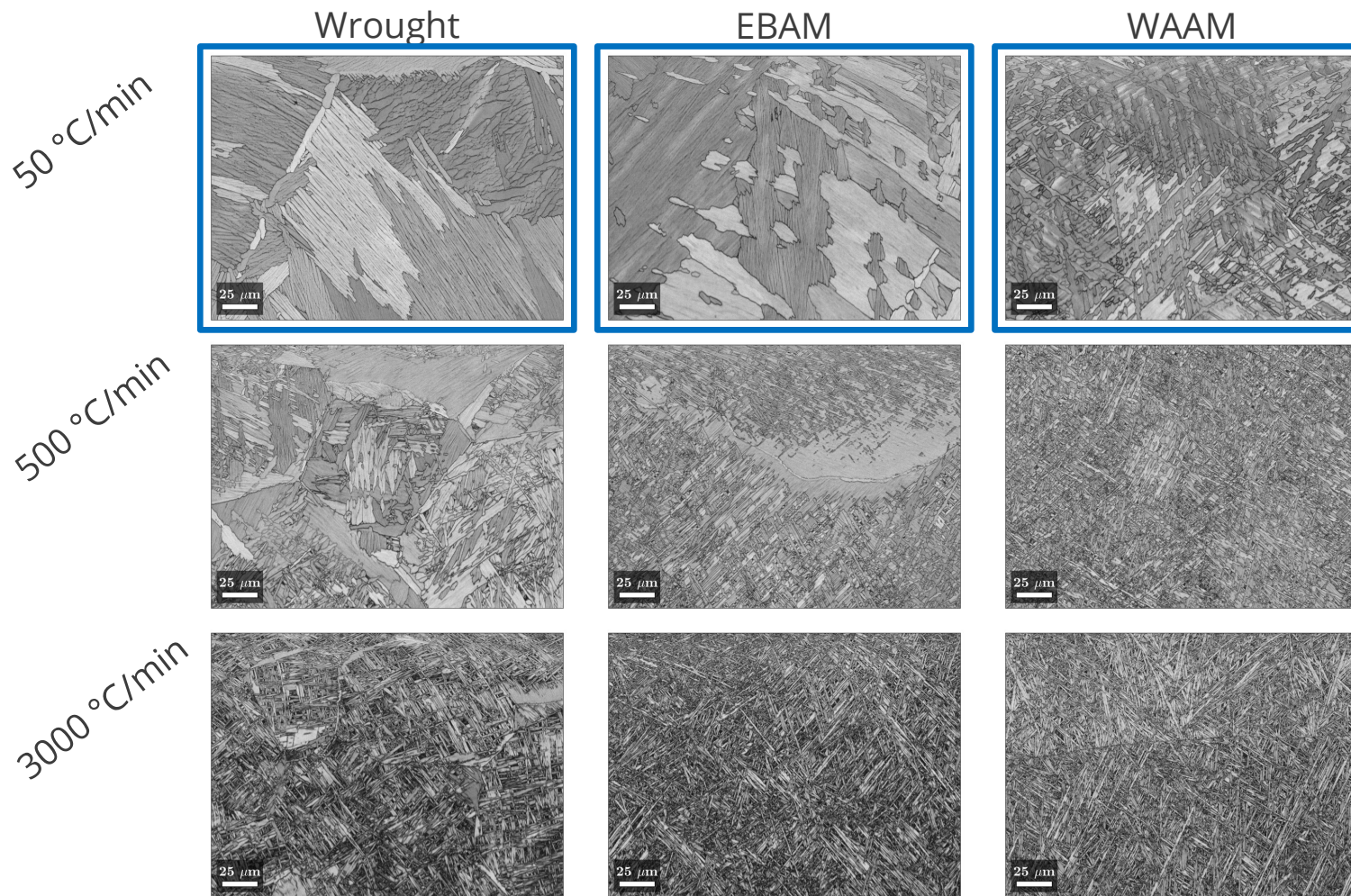
↓  
Acicular  $\alpha'$





13

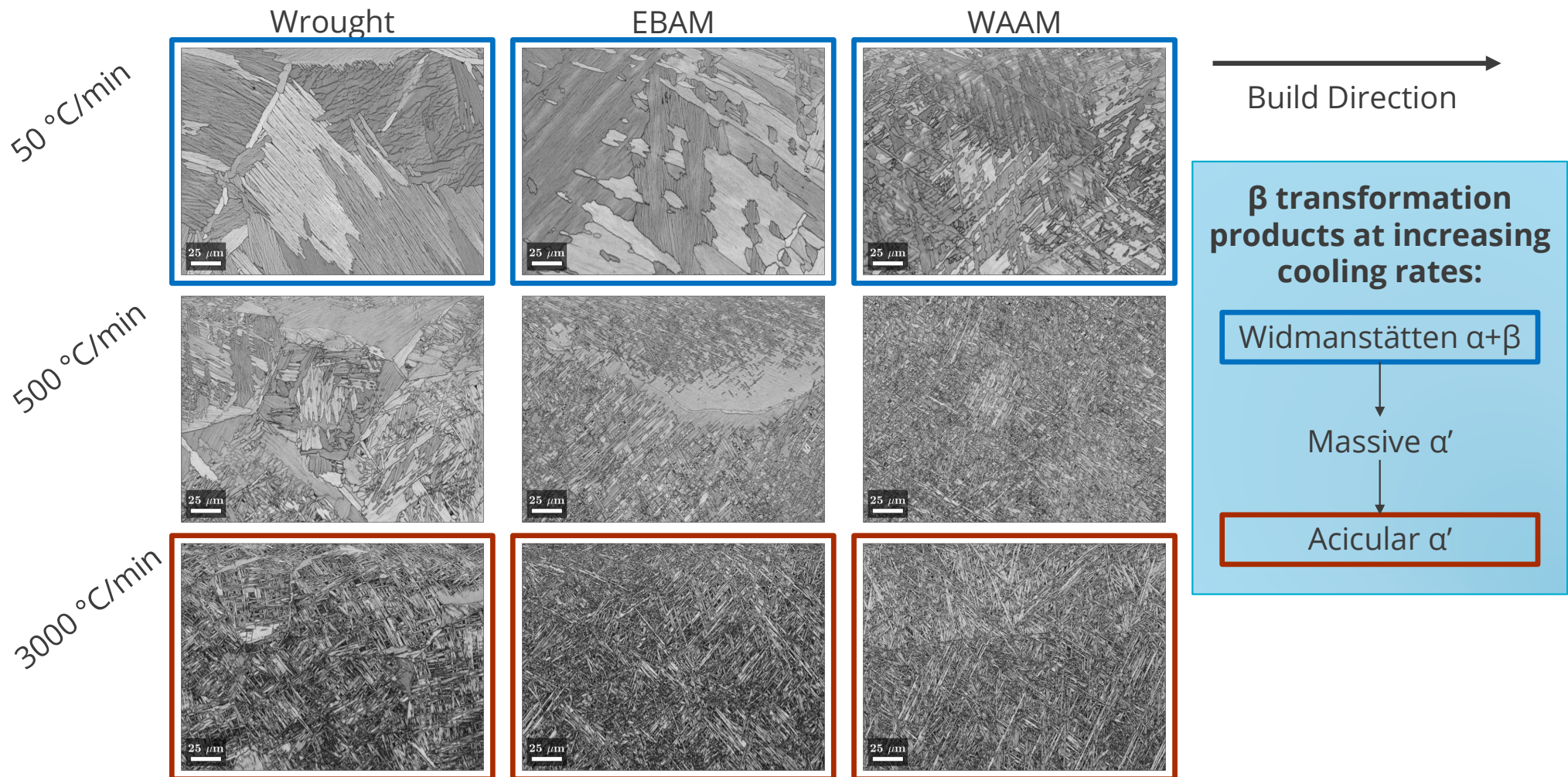
# Microstructure was sensitive to both cooling rate and processes





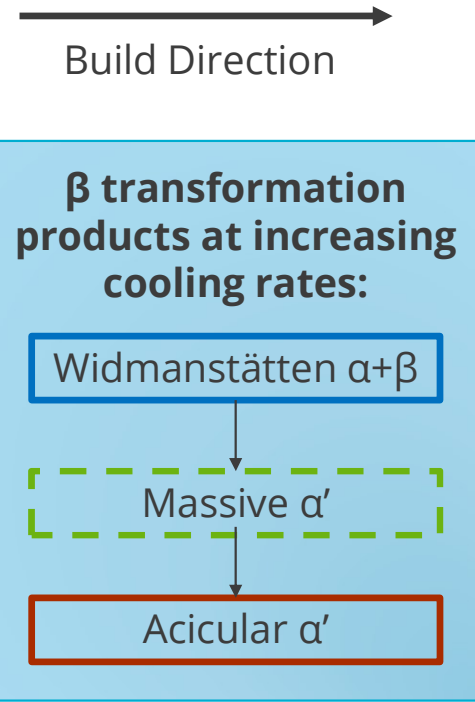
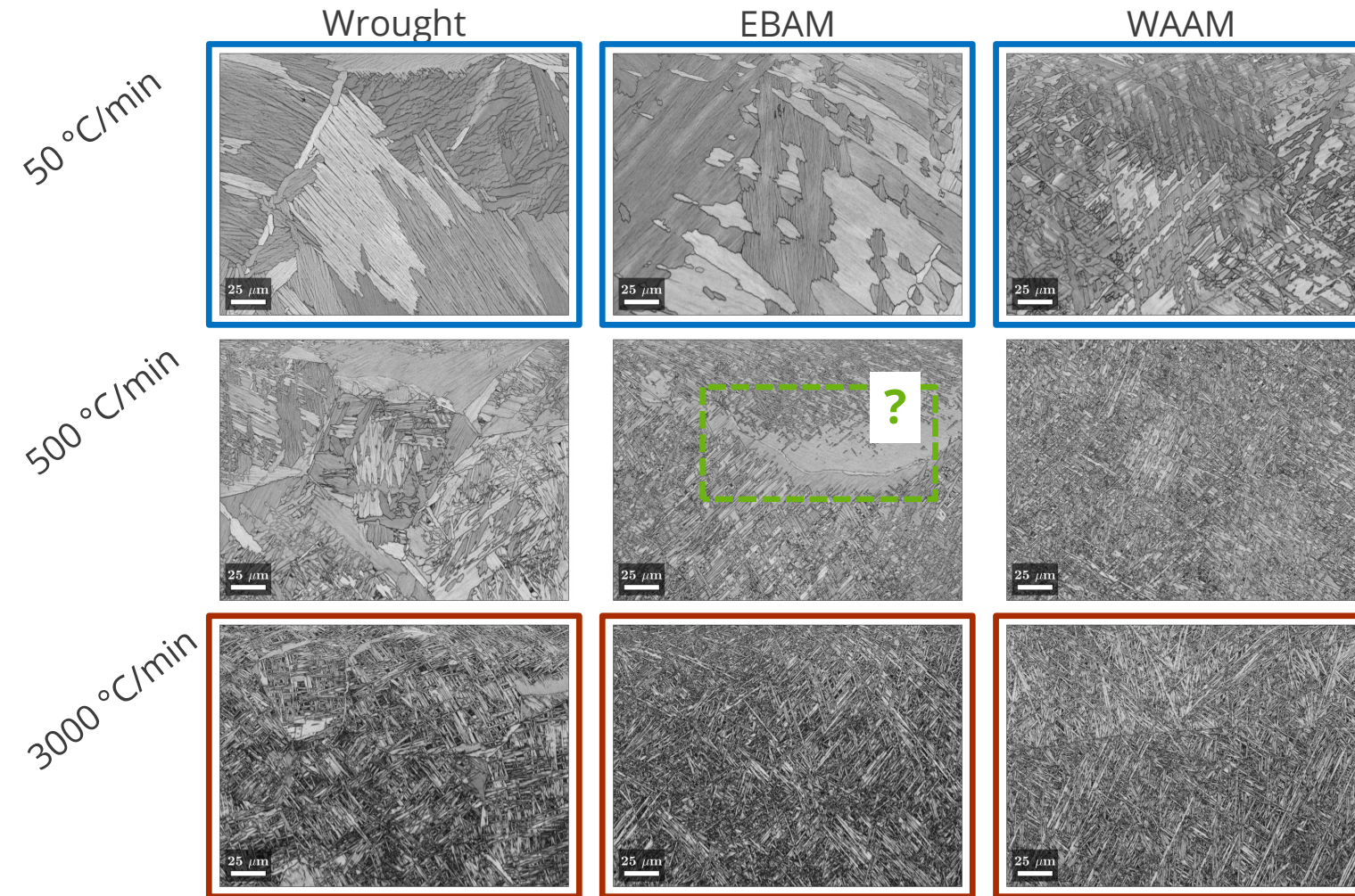
14

# Microstructure was sensitive to both cooling rate and processes





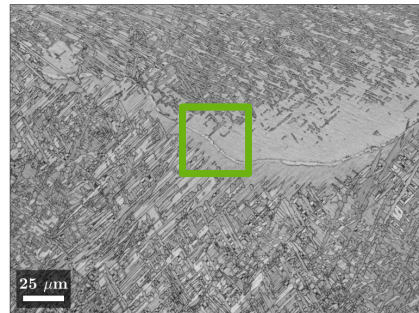
# Microstructure was sensitive to both cooling rate and processes



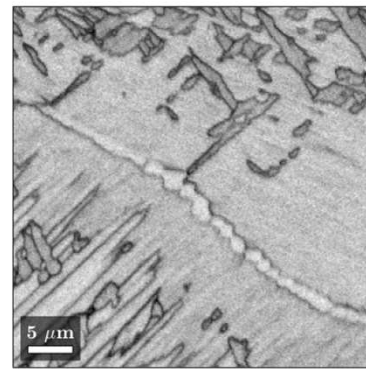


# Parent $\beta$ grain boundaries partially control microstructure dev

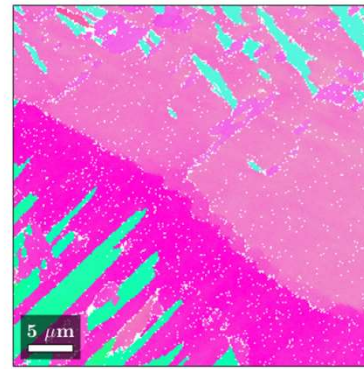
HCP-Ti ( $\alpha/\alpha'$ ) EBSD analysis



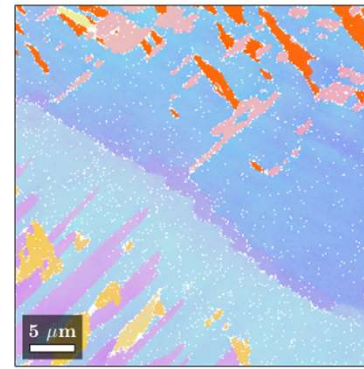
Build Direction  $\rightarrow$



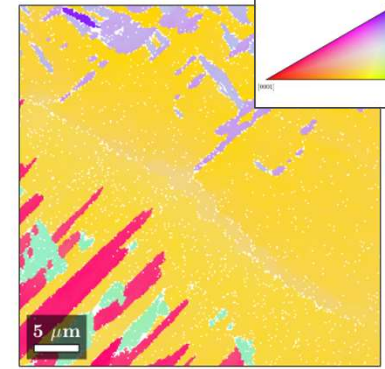
BC



IPF-X

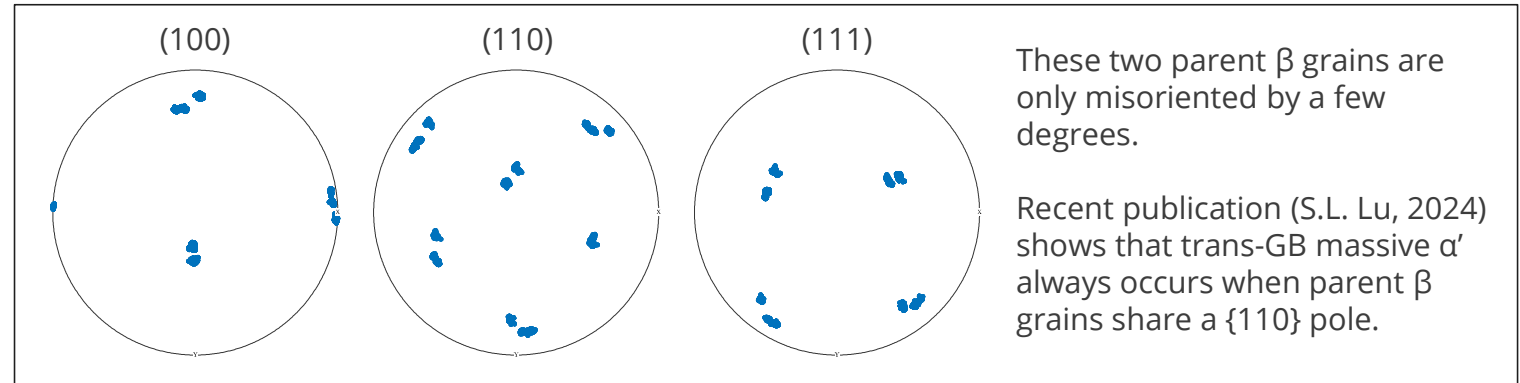


IPF-Y



IPF-Z

FCC-Ti ( $\beta$ ) grains reconstructed with OR:  $(110) \parallel (0001) \quad [-11-1] \parallel [2-1-10]$





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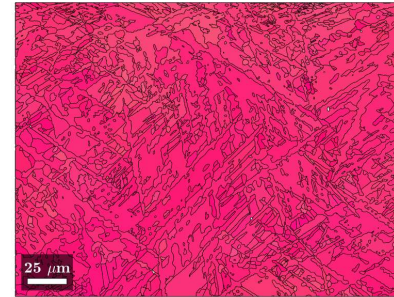
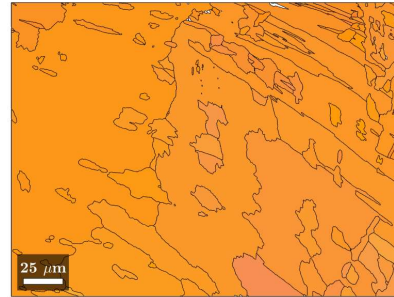
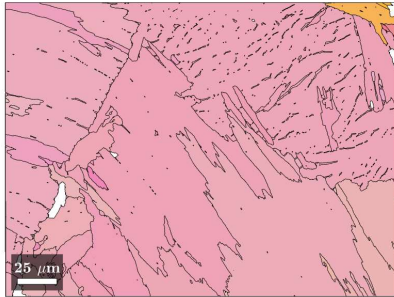
# Parent $\beta$ grain boundaries partially control microstructure dev

Wrought

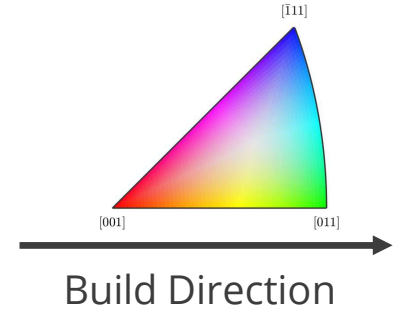
EBAM

WAAM

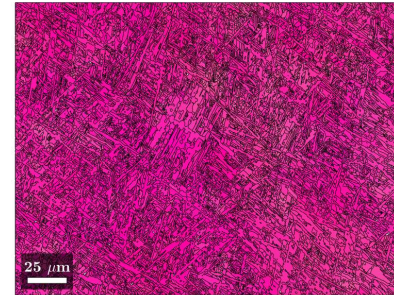
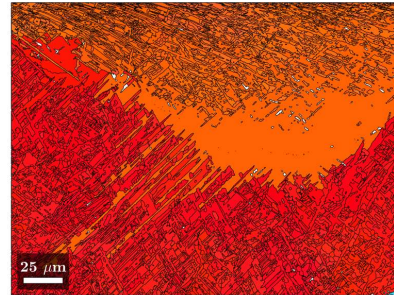
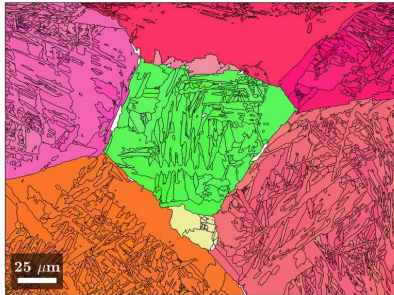
50 °C/min



FCC-Ti ( $\beta$ ) parent grains  
reconstructed with OR:  
(110) || (0001) [-11-1] || [2-1-10]  
Alpha substructure is overlaid.

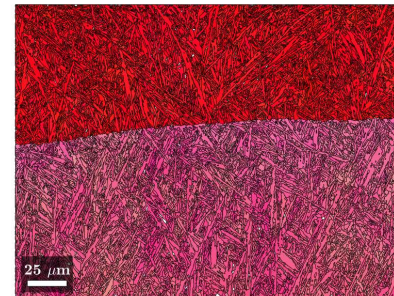
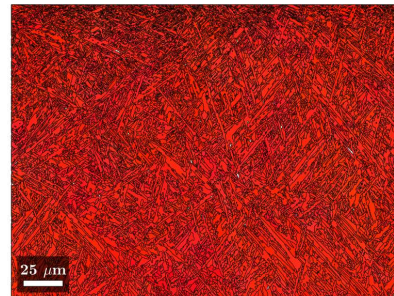
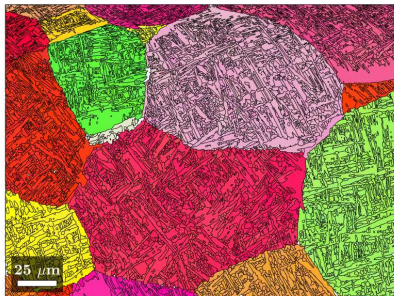


500 °C/min



Build Direction

3000 °C/min

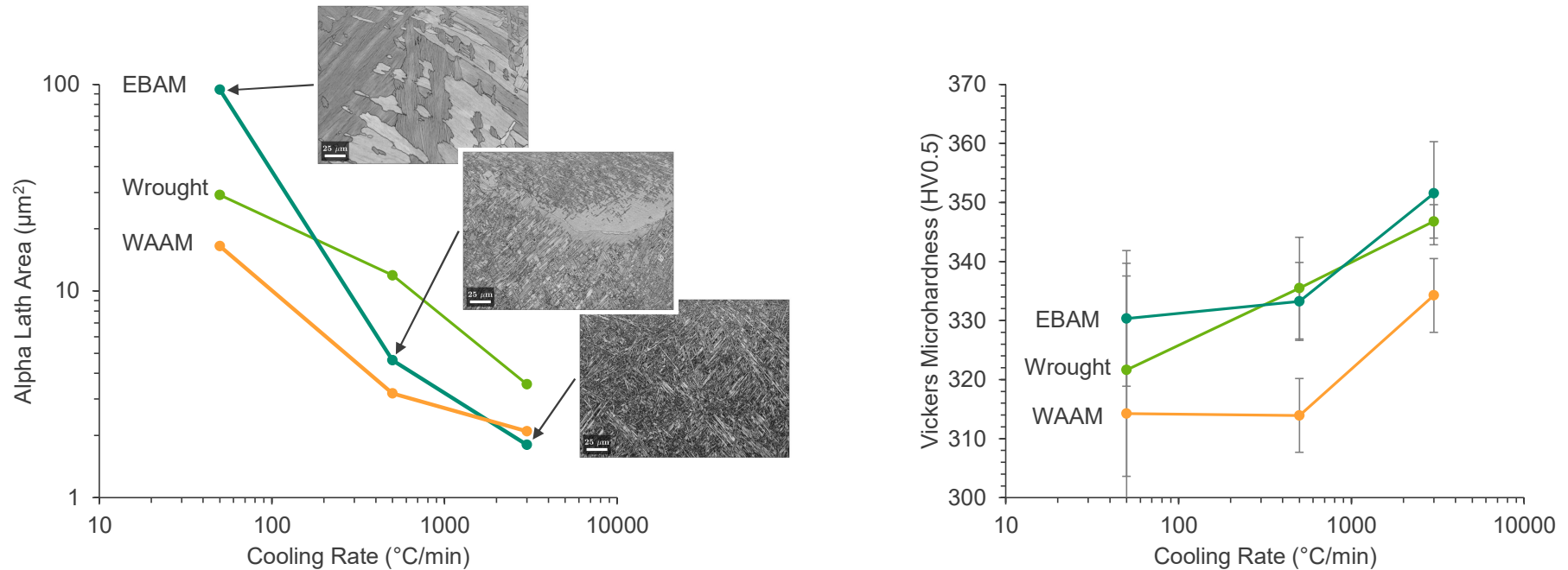


This effect is  
intensified by the  
large, columnar, and  
highly textured  
parent  $\beta$  grains in  
the EBAM and  
WAAM builds!



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## Cooling rate affected transformed $\beta$ size and thus the mechanical properties



Although an upward trend in hardness with increasing CR is obvious, a deeper study of microstructure-properties relationships is needed. This work is ongoing.



## Conclusions

- We simulated post-build thermal treatments to understand solid state phase transformations on cooling from above the  $\beta$  transus
- Instrumented quench dilatometry gave insight into transformation temperatures
- Known trends for microstructure development were confirmed
  - i.e., Widmanstätten  $\alpha+\beta \rightarrow$  massive  $\alpha' \rightarrow$  acicular  $\alpha'$
  - Parent  $\beta$  boundaries appear to play an additional role
- Process-induced chemical variations remain important for AM Ti-6Al-4V
- These data will be used to tailor heat treatments for improved performance in service. Future work will clarify microstructure-property relationships.

**Chris Finfrock, [cfinfro@sandia.gov](mailto:cfinfro@sandia.gov)**

Jessica Buckner, Jonathan Pegues, Hannah Sims, Jack Herrmann, Anthony Marcon