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Processing of Fe-Co Soft Ferromagnetic Alloys using Laser Engineered Net Shaping (LENS)

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Characterization of the Fe-Co-1.5V soft ferromagnetic alloy processed by Laser Engineered Net Shaping (LENS), in *Additive Manufacturing*, 2018

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The metallurgy of Fe-Co alloys

Equiatomic or near-equiatomic Fe-Co alloys that undergo a γ-FCC → α-BCC→ α_2 (B_2) transformations.

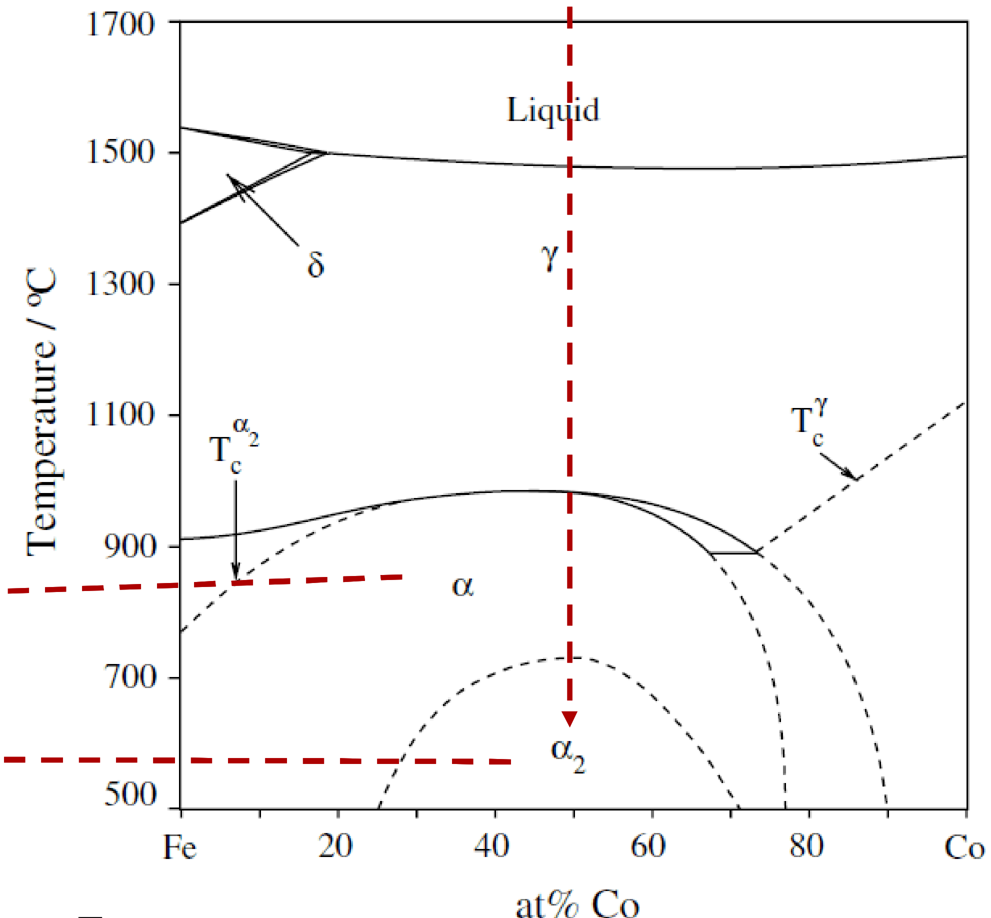
- Poor composition-driven workability, binary Fe-Co difficult to process.
- Commercialized as Fe-Co-2V (Hiperco®) in bar, sheet, strip, coil, and rod forms.

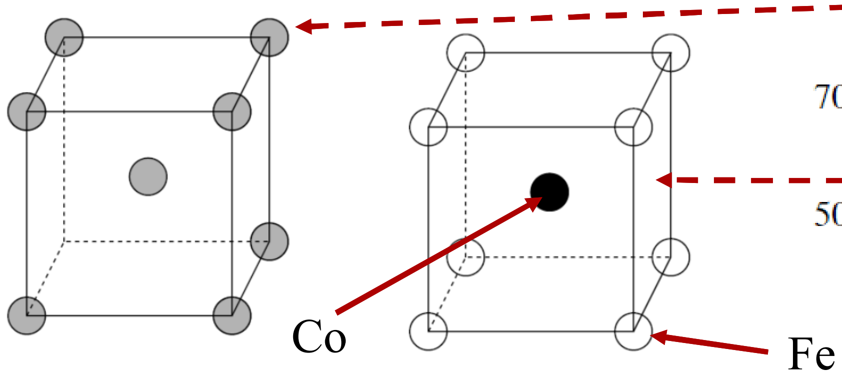
Hiperco® is a tradename of Carpenter Technologies, Reading, PA.

Excellent magnetic properties:

- Highest saturation induction of all engineering soft ferromagnetic alloys
- High curie temperature (> 900 °C)
- High permeability
- Low core loss

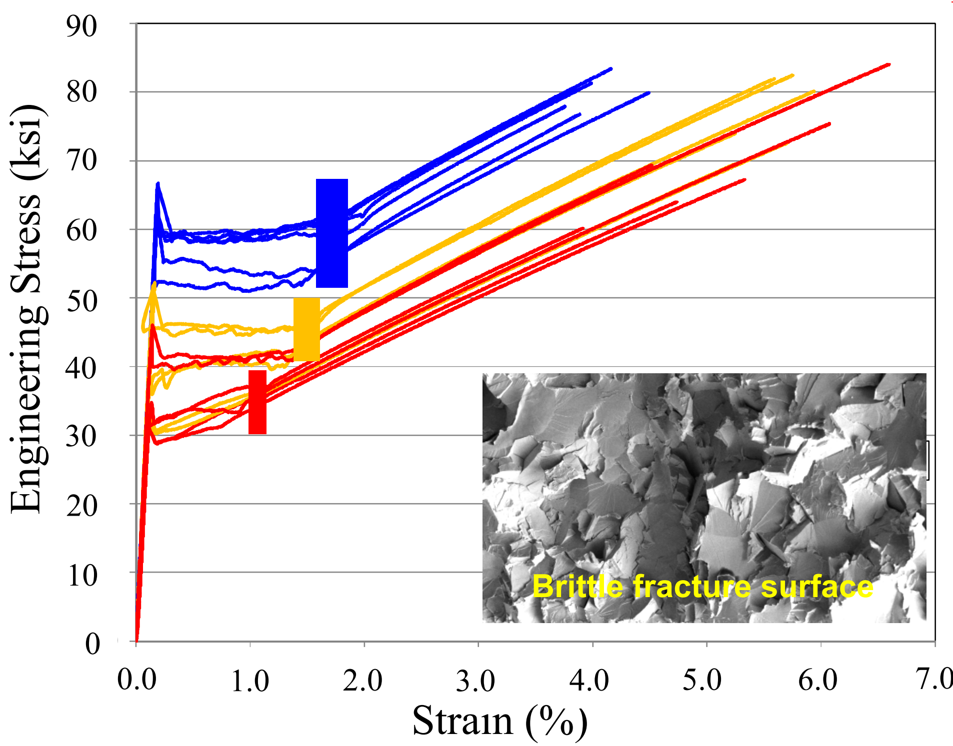
Atomic ordering is a big issue!

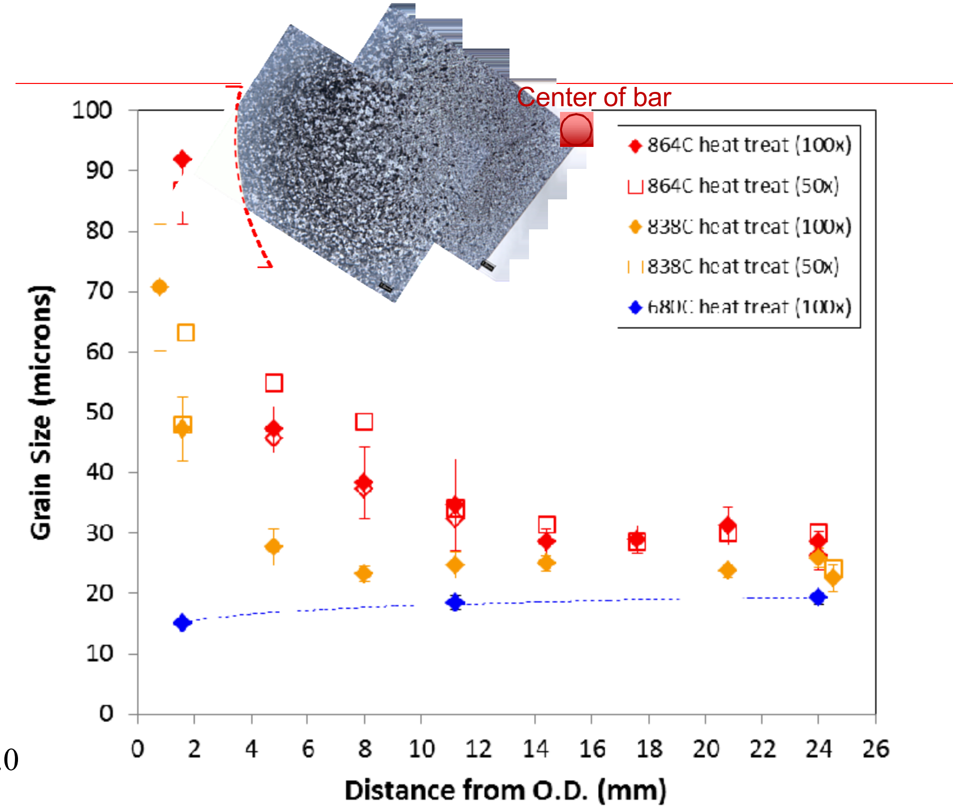




What are the problems with Hiperco?

- Forged (conventional) Hiperco is weak, brittle, and has inhomogeneous microstructures.





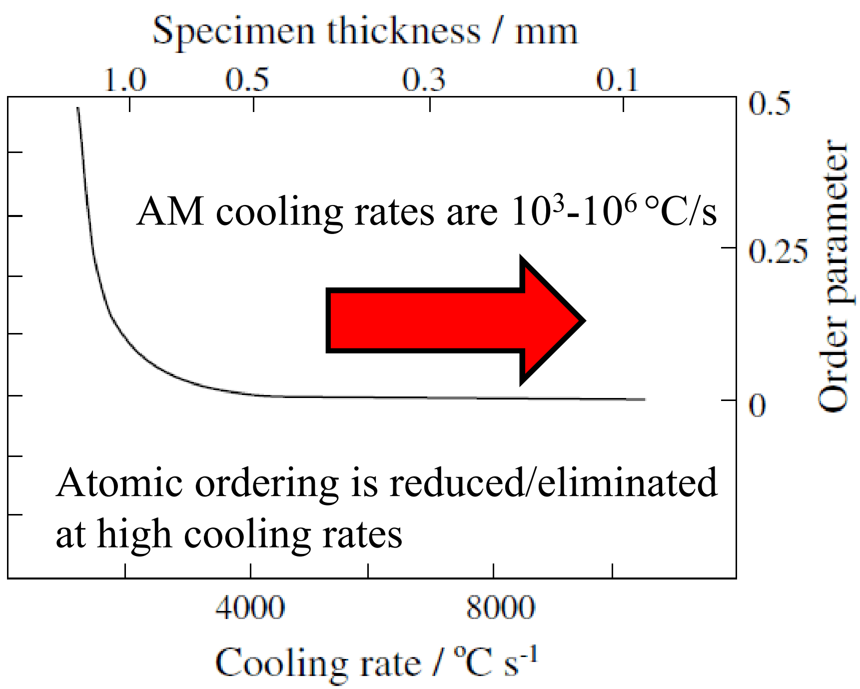
- Goal: achieve uniform microstructures with controlled atomic ordering.

  - Is Additive Manufacturing the processing solution?

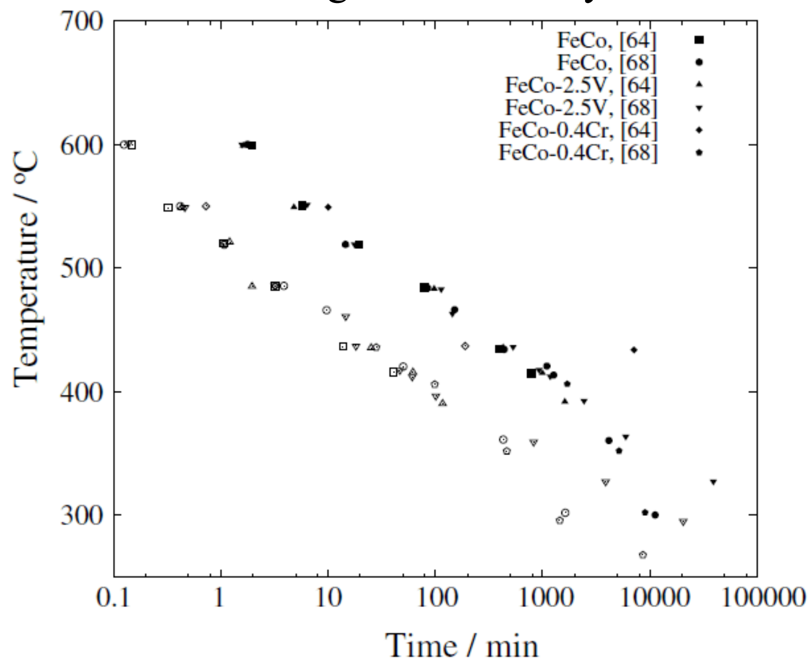
Additive manufacturing: the processing solution?

Hypothesis: The unique thermal history of layer-by-layer AM will inhibit atomic ordering in a controlled and predictable way.

Through AM, avoid workability issues that arise in conventional thermomechanical processes through a solidification-based processing solution.

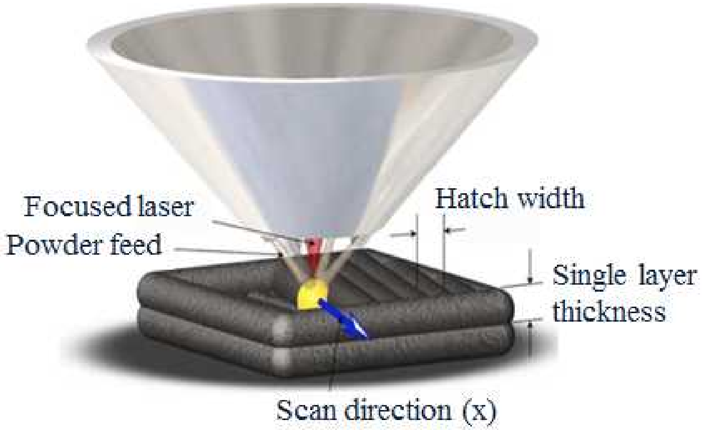


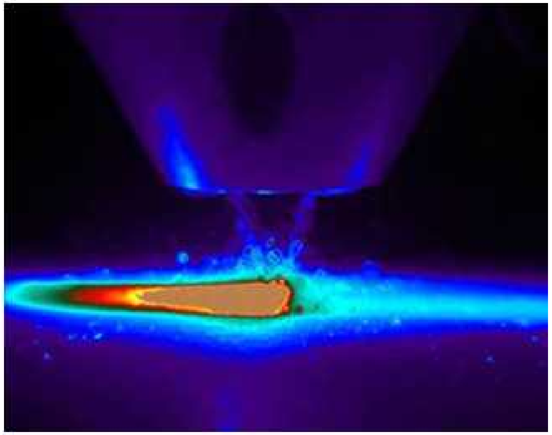
Time at temperature for 50% ordering in Fe-Co alloys.

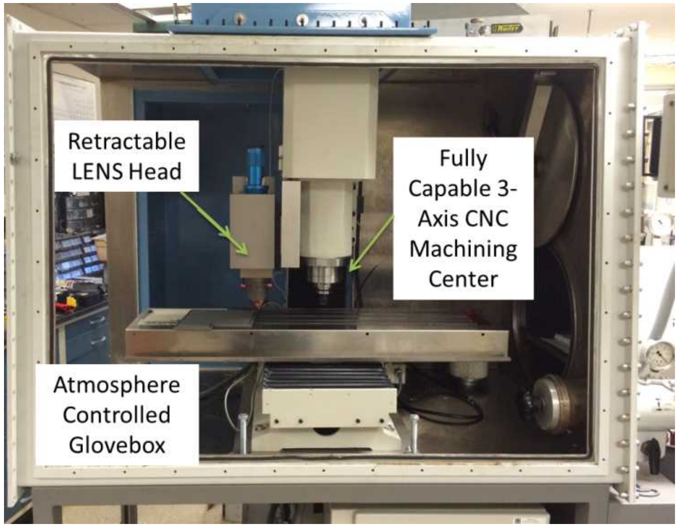


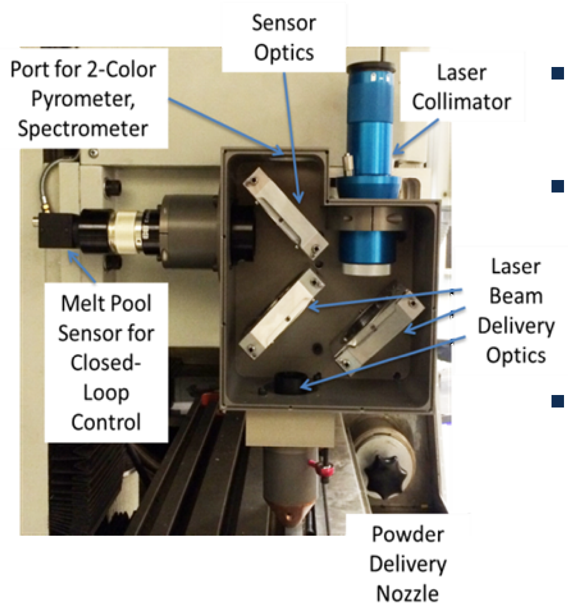
Will AM be an **enabling** process for these materials?

Laser Engineered Net Shaping (LENS)









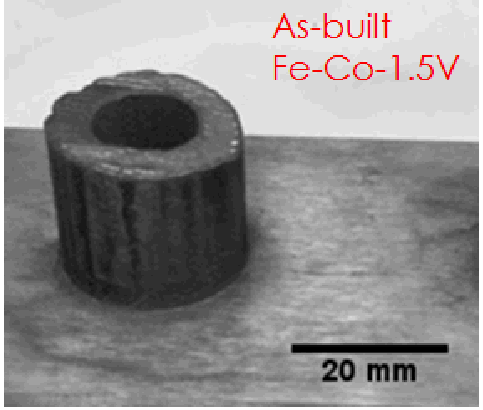
- Laboratory-scale LENS in Tormach CNC 770 frame.
- YLS-2000 Laser from IPG Photonics with 2 kW maximum output at 1064 nm.
- Control the powder feed through feed wheel and carrier gas (independently) to fluidize the powder.

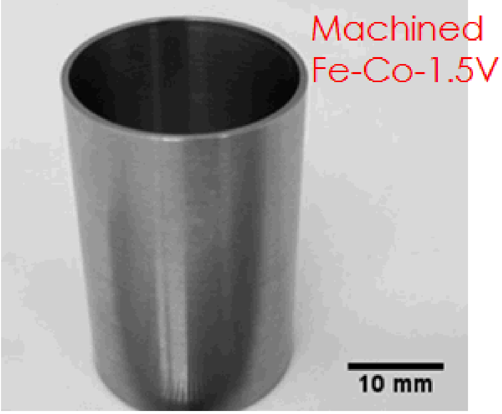
LENS processed soft ferromagnetic alloys

Sandia National Laboratories

<u>Conditions</u>

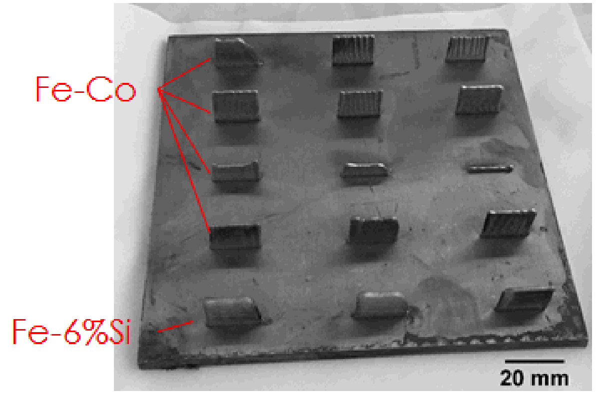
Laser power = 150-450 W
Build speed = 150-600 mm/min
Interlayer interval time = 0.3-10 s

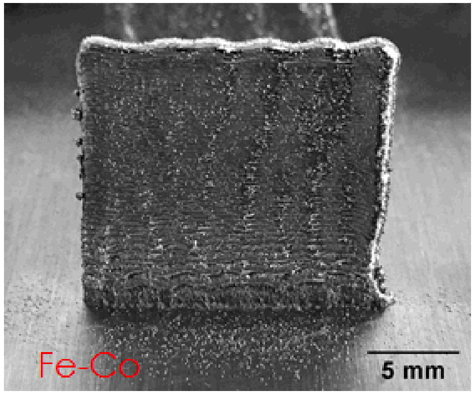




<u>Feature Result</u>

Fe-Co ***<u>and</u>*** Fe-Si alloys were processed *via* LENS – including Fe-Co-1.5V, Fe-Co, and Fe-6%Si.





Thin walls for measuring ordering

Varied laser power, build speed, and time between subsequent layers (interlayer interval time) to:

1. Control the degree of retained heat within LENS thin walls.
2. Impose near order of magnitude variation in predicted cooling rate.

Rosenthal Model: $\frac{dT}{dt} \cong \frac{\kappa v_b}{\alpha Q}(T_m - T_o)^2$

Processing Parameters				Output Parameters
Specimen	Laser Power, Q (W)	Build Speed, v_b (mm/s)	Interlayer Interval Time, t_l (s)	Rosenthal Predicted Cooling Rate (K/s)
1	150	3	0.3	3.6 E3
2	150	7	0.3	9.5 E3
3	300	4	0.3	2.7 E3
4	300	8	0.3	5.9 E3
5	450	10	0.3	4.8 E3
6	150	3	10	3.6 E3
7	150	7	10	9.5 E3
8	300	3	10	1.8 E3
9	300	4	10	2.7 E3
10	300	8	10	5.9 E3

Thin walls for measuring ordering

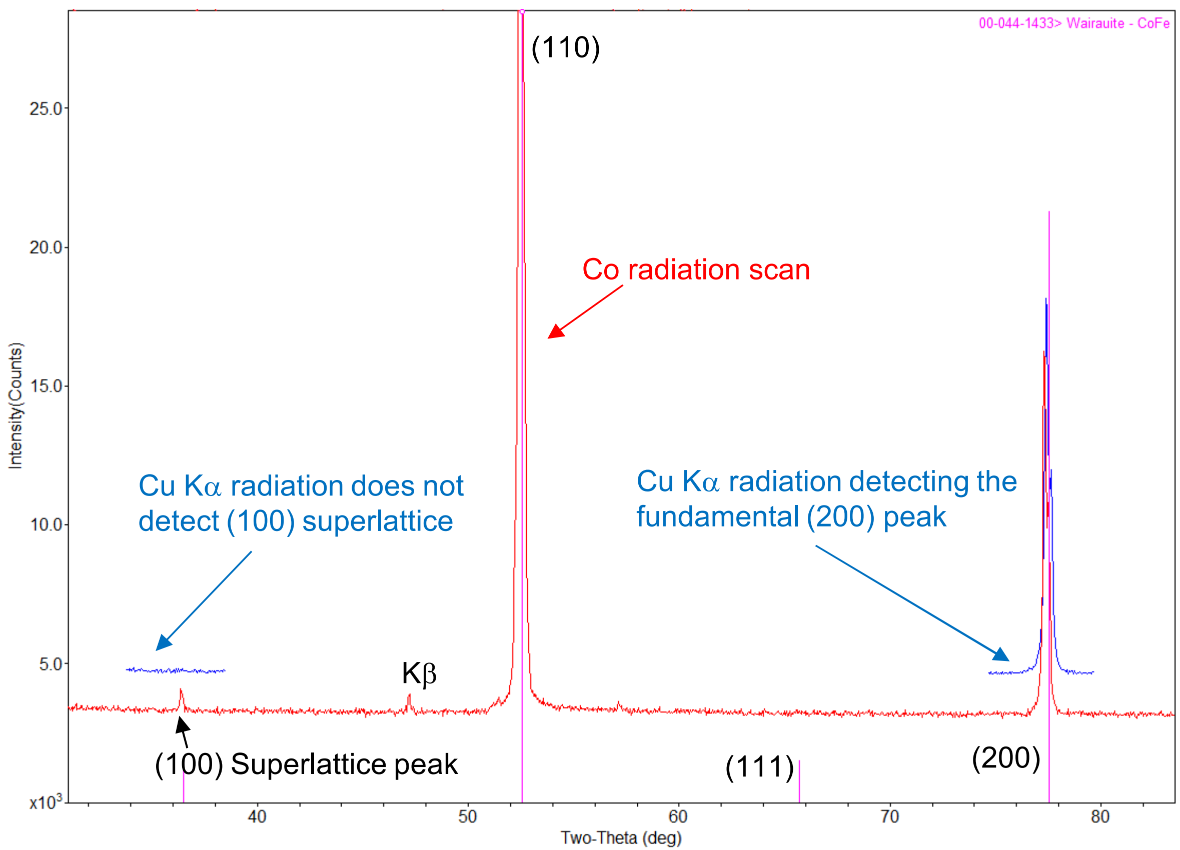
Characterization of ordering:

- Tracked (100) superlattice peak count intensity relative to (200).
- Ratios were normalized to an annealed condition for ***relative*** ordering.
- Used Cobalt X-ray radiation to characterize samples - Cobalt source required to see superlattice!

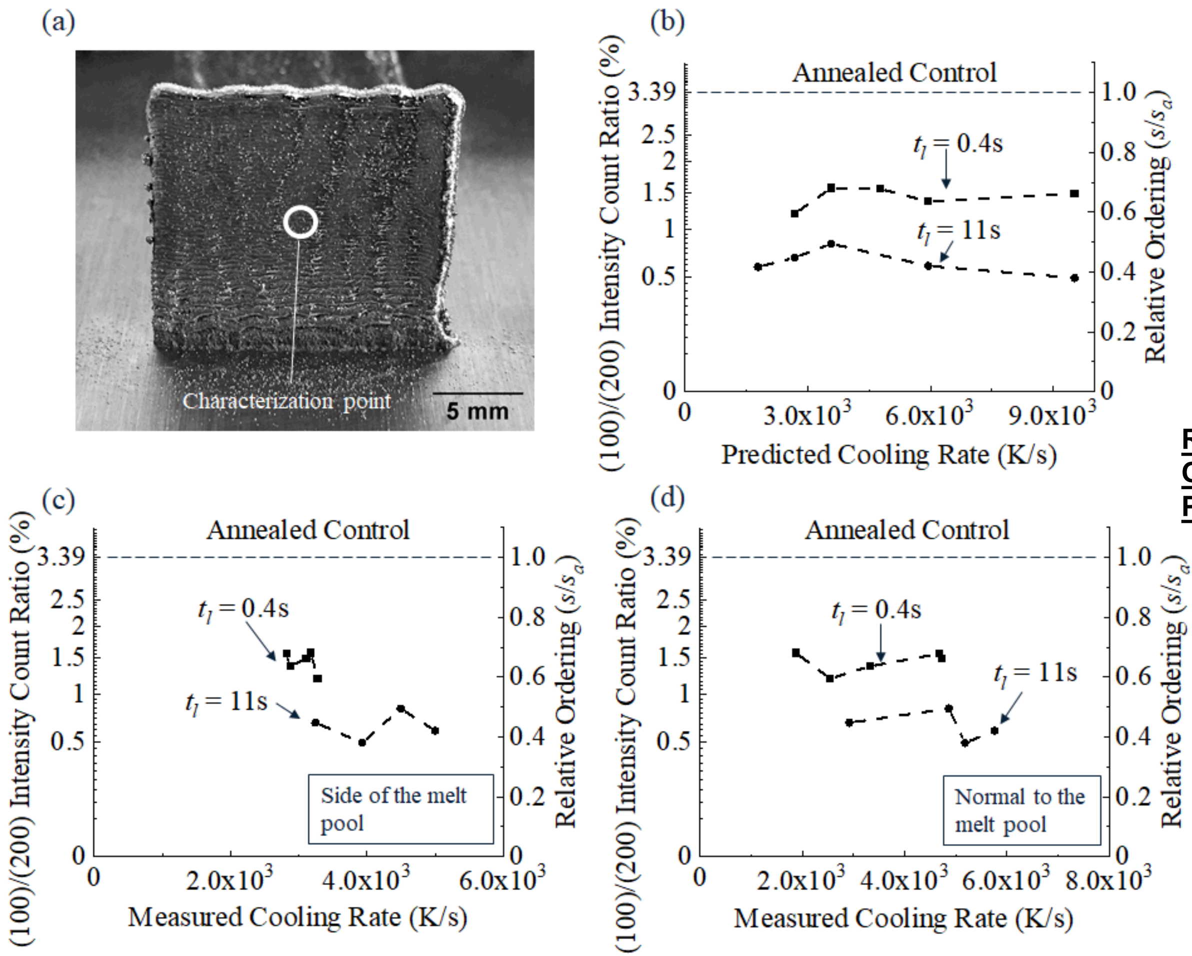
Relative Ordering Parameter

$$\frac{s}{s_a} = \sqrt{\frac{\frac{I_{100}}{I_{200}}}{\left(\frac{I_{100}}{I_{200}}\right)_a}}$$

Connect the atomic ordering parameters with the LENS processing conditions



Reduced ordering via LENS!

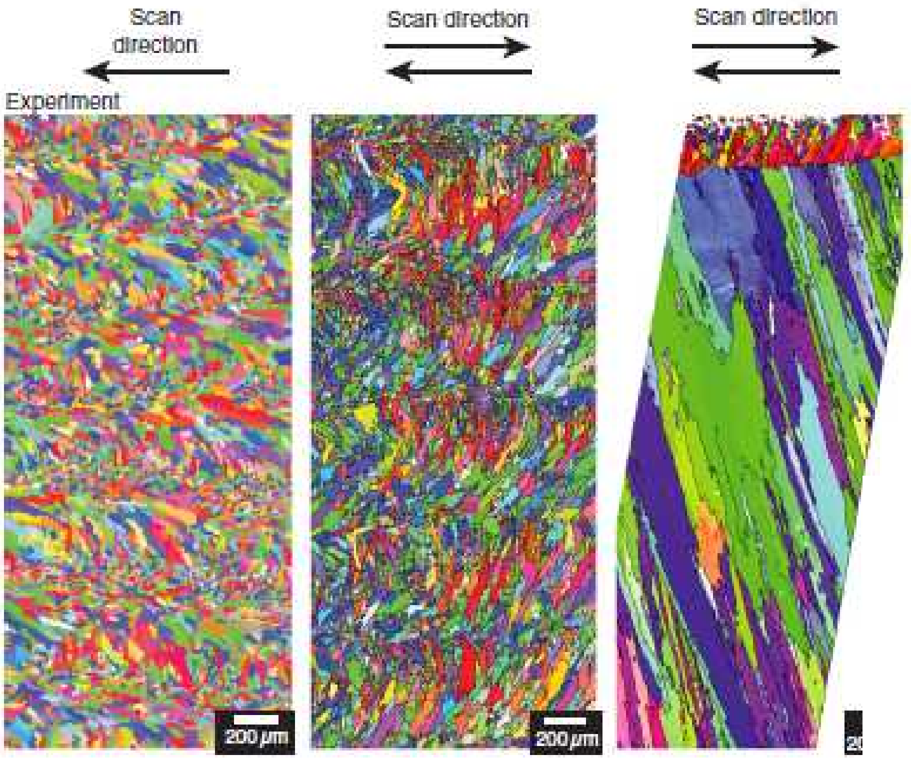


Relative Ordering Parameter

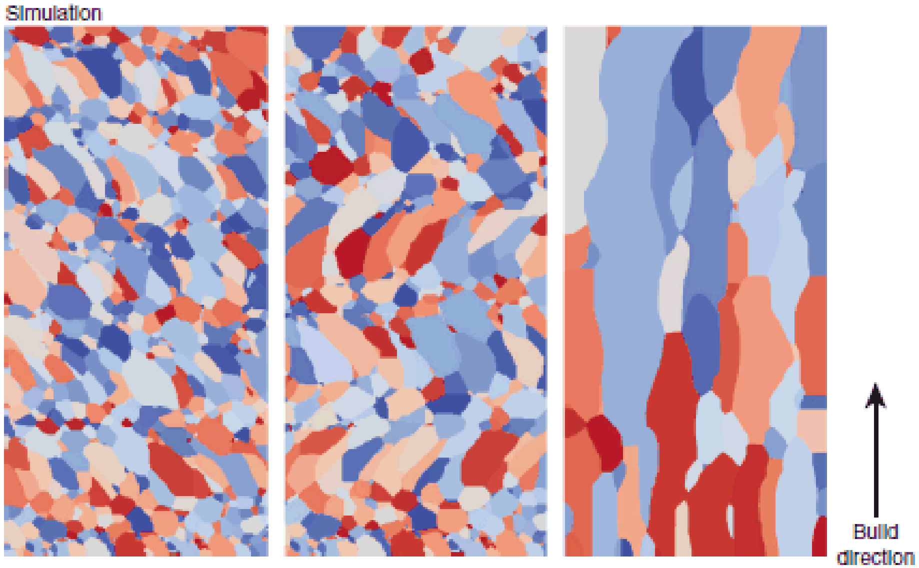
$$\frac{s}{s_a} = \sqrt{\frac{\frac{I_{100}}{I_{200}}}{\left(\frac{I_{100}}{I_{200}}\right)_a}}$$

- Hypothesis validated: X-ray measurements suggest AM samples 40-70% ordered compared to annealed sample.
- The interlayer interval time had significant effects on ordering. With increased interval time, ordering dropped due to increased cooling rate and sharper thermal gradient.

Columnar-type structures are common in LENS



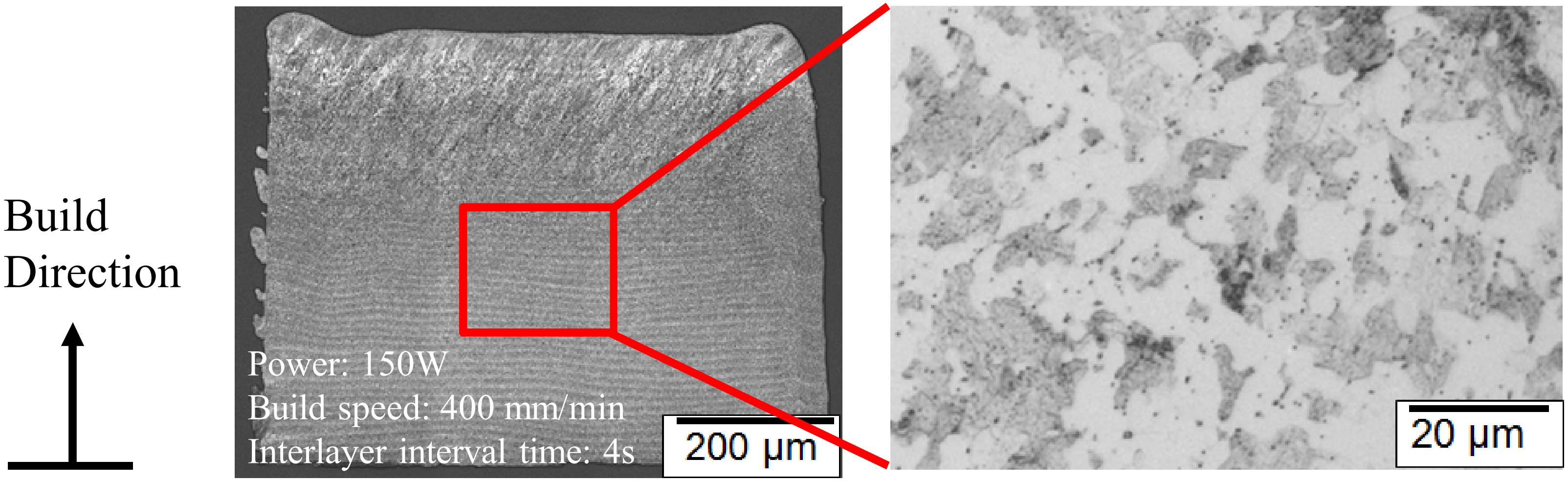
Columnar grain morphologies often observed in materials processed by LENS/AM.

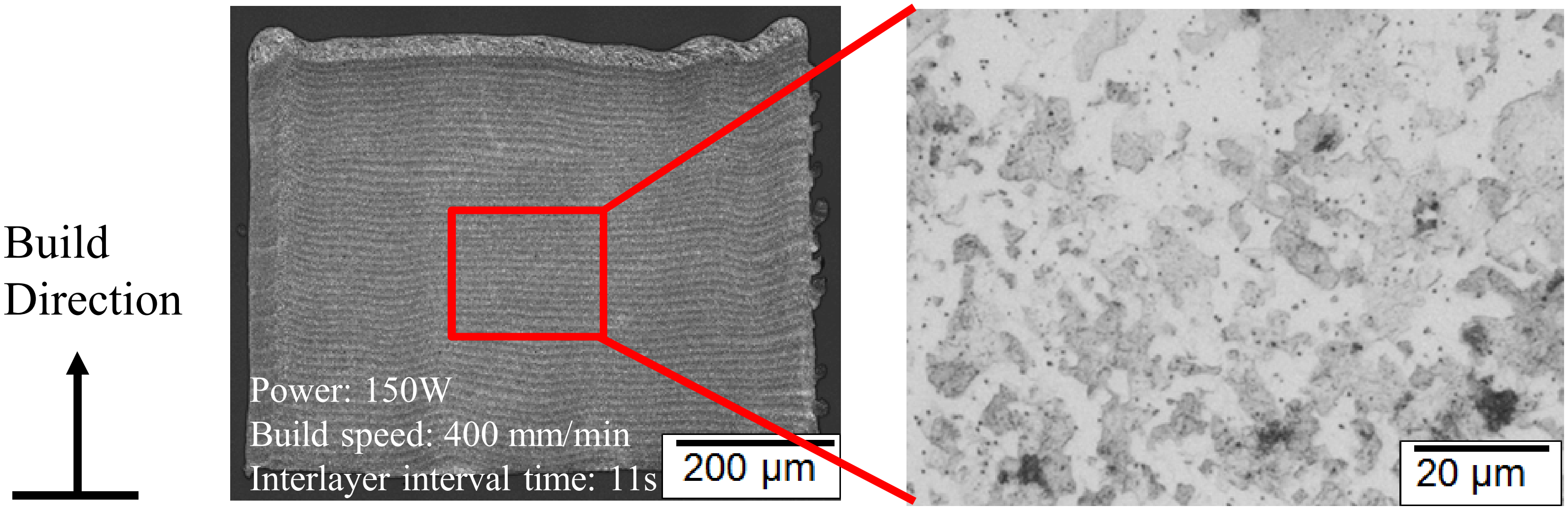


a) Build 1: Low-power unidirectional
b) Build 2: Low-power bidirectional
c) Build 3: High-power bidirectional

Similar structures have been noted in simulations - Theron Rodgers, et al., Comp. Mater. Sci., 2015.

Fine equiaxed grain structure in as-built Fe-Co-1.5V thin walls



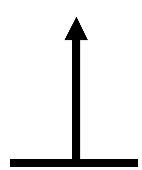


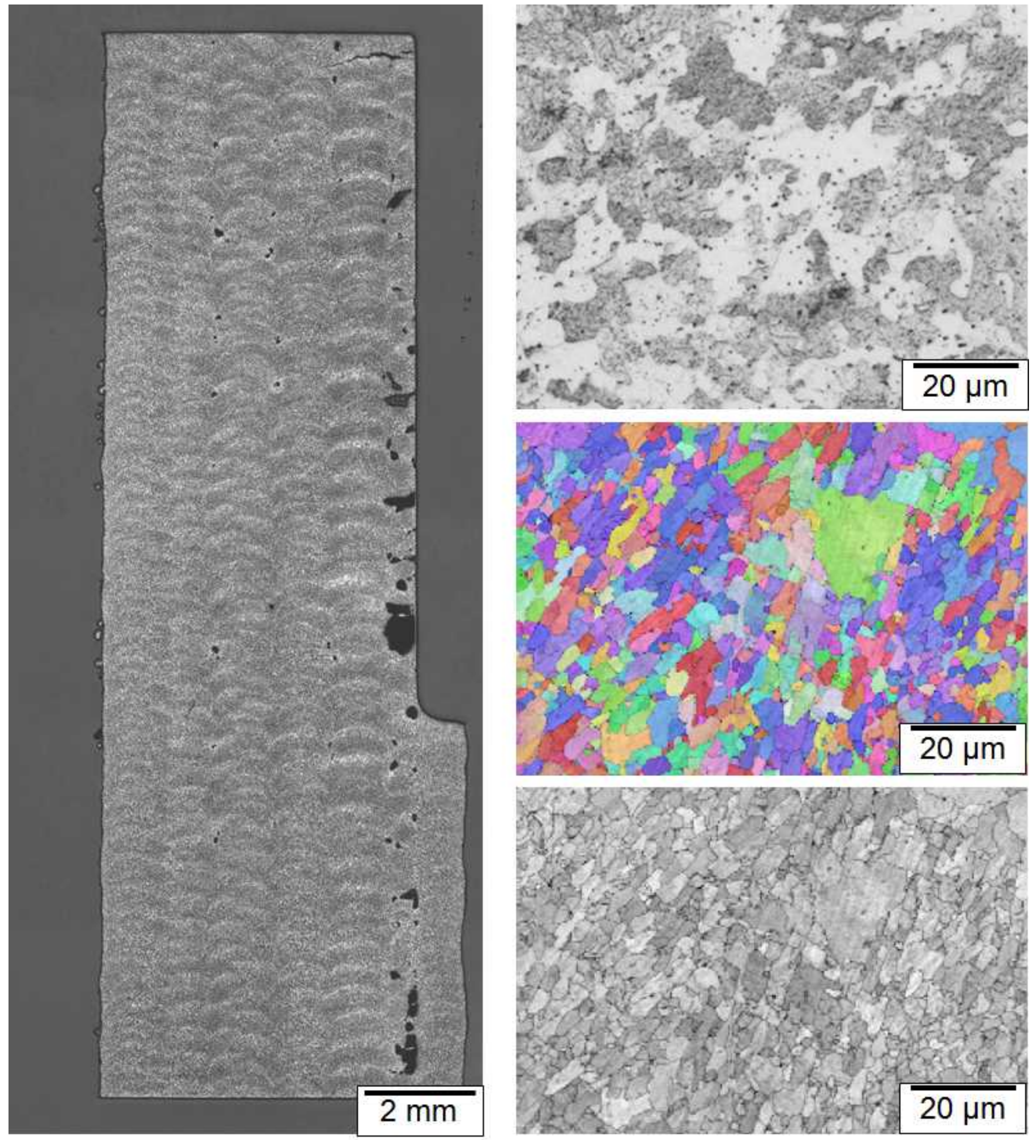
Similar structure in bulk cylinders

- Fine equiaxed grains throughout the cylinder.
- Crystallographic texture was weak (near-random).

Can we compare with microstructure predictions using conventional solidification theory?

Build Direction

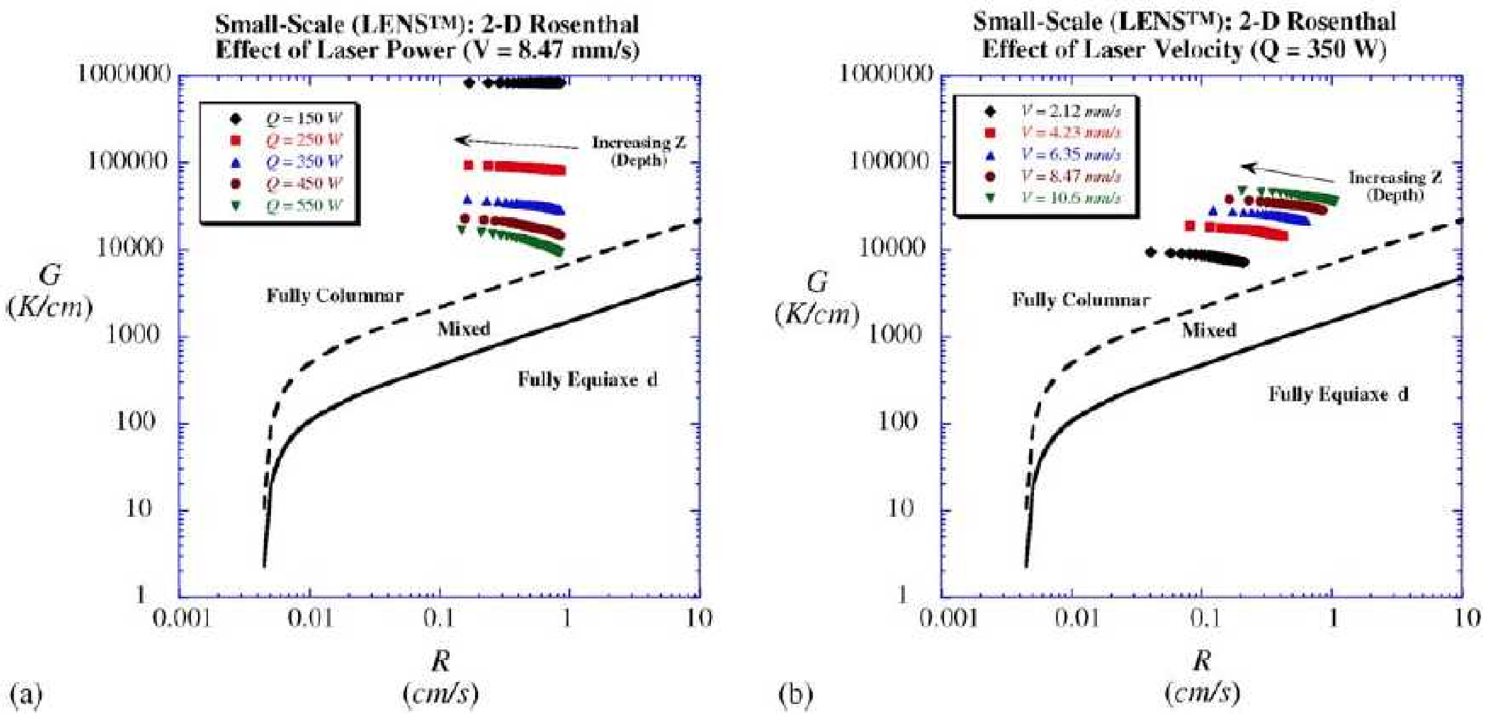




Analysis using columnar-to-equiaxed transition (CET) model

- First-order analytical analysis of solidification microstructures applied to conventional castings.

- Utilized in some AM literature (ORNL, Wright State/AFRL, CMU).



CET model analysis

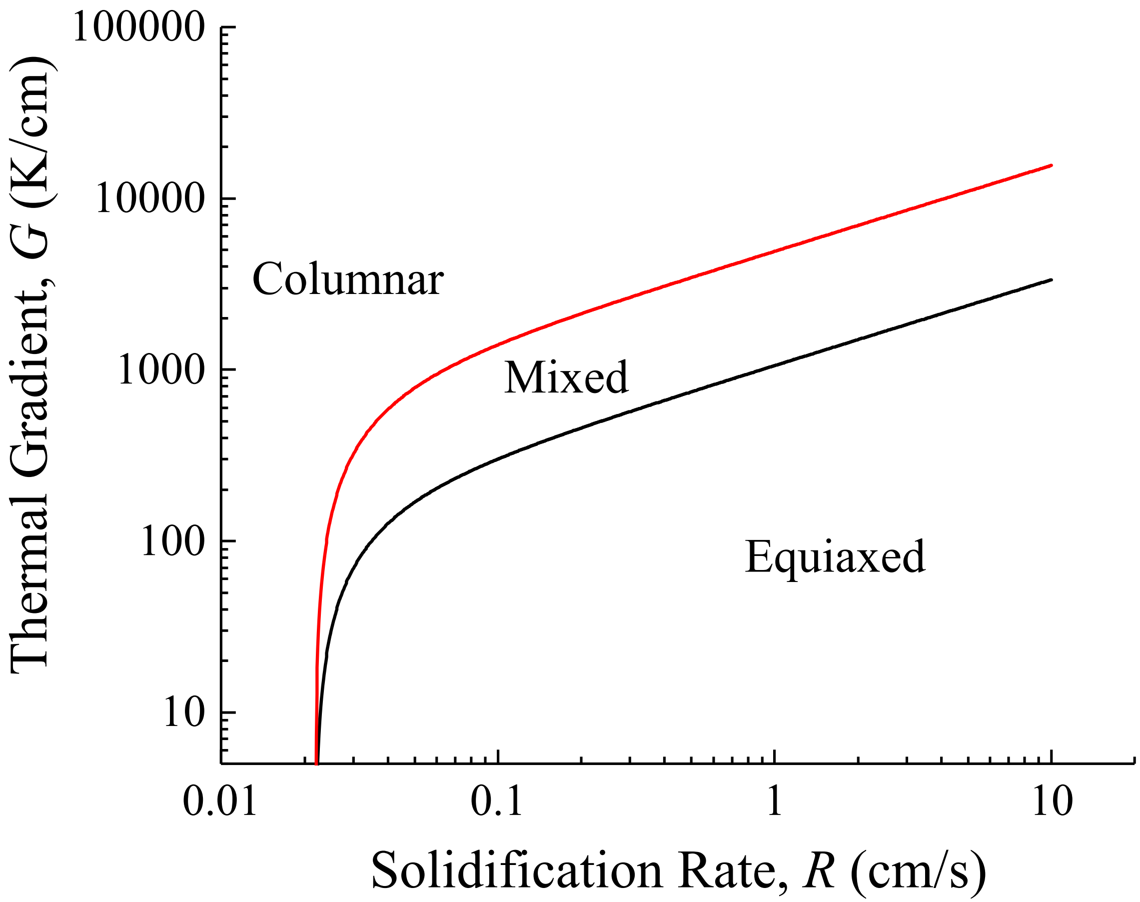
$$G < 0.617\left(N_o\right)^{1/3}\left(1-\frac{(\Delta T_N)^3}{(\Delta T_c)^3}\right)(\Delta T_c)$$ Fully Equiaxed Condition

$$G > 0.617\left(100N_o\right)^{1/3}\left(1-\frac{(\Delta T_N)^3}{(\Delta T_c)^3}\right)(\Delta T_c)$$ Fully Columnar Condition

ΔT_N = undercooling at the heterogeneous nucleation temperature

ΔT_c = dendrite tip undercooling

N_o = number of heterogeneous nucleation sites



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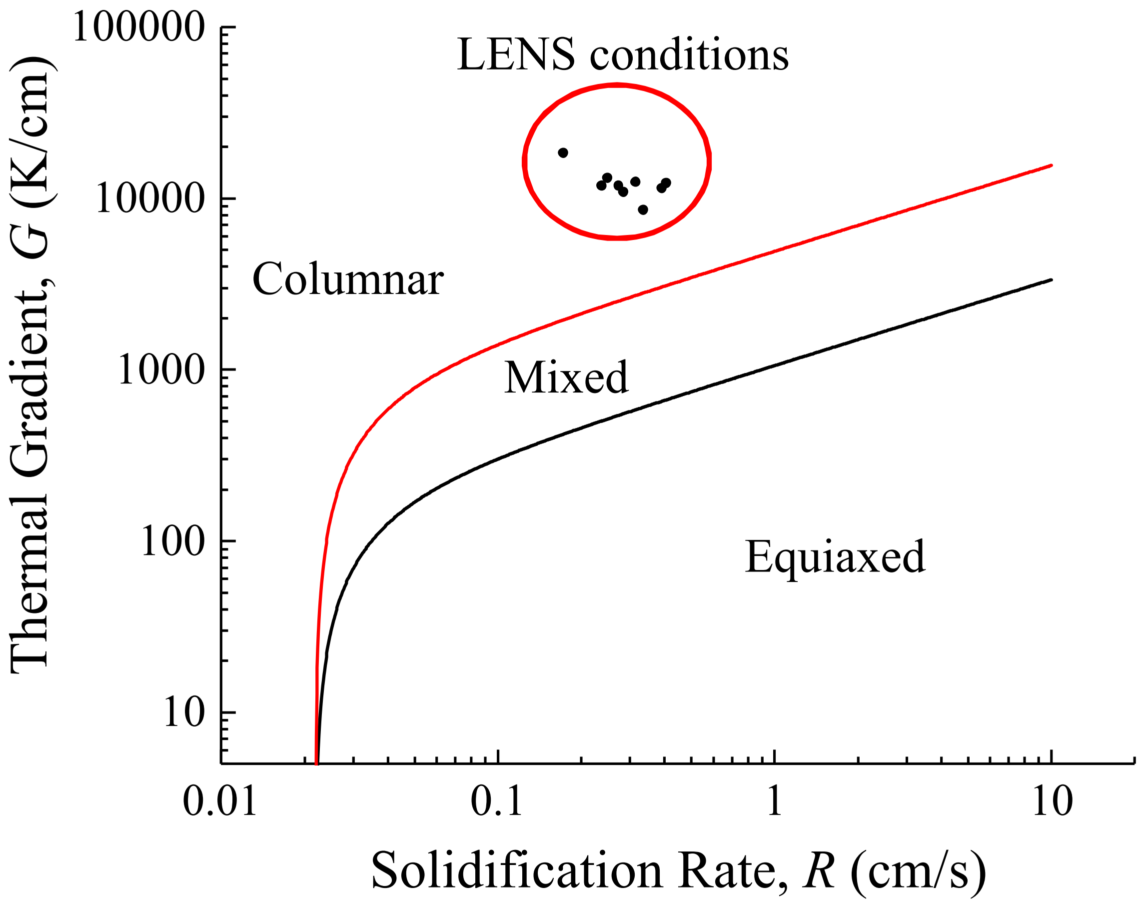


ΔT_N = undercooling at the heterogeneous nucleation temperature

ΔT_c = dendrite tip undercooling

N_o = number of heterogeneous nucleation sites

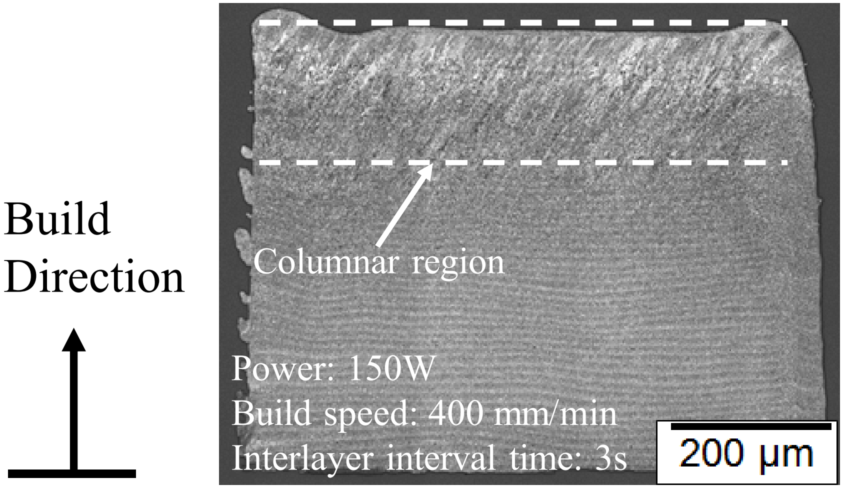
Measured thermal conditions for thin walls were in the 'fully columnar' regime of Hunt's CET theory!

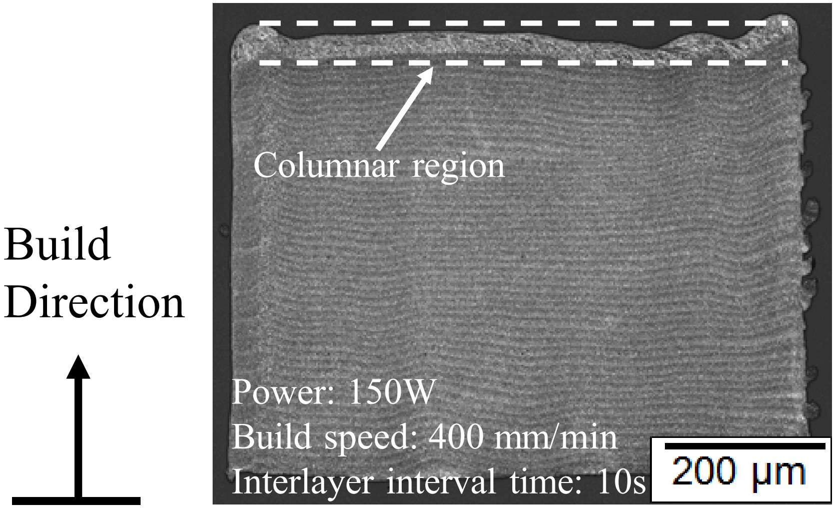
Reasons for the discrepancy?

1. Cyclic thermal conditions of AM:
   a. Reduced thermal gradient or increase in solidification rate in subsequent layers
   b. Repetitive solid-state γFCC-αBCC phase transformations
   c. Double recalescence

2. High density of inoculants (e.g., $(Fe,Co)V_3$ particles, oxides inclusions).

Layer-by-layer reheating/remelting may induce change





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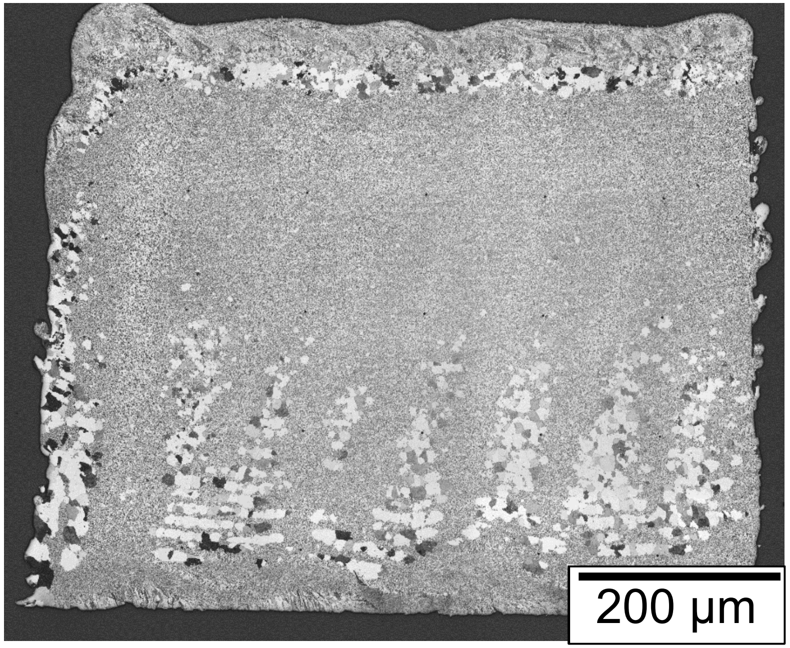
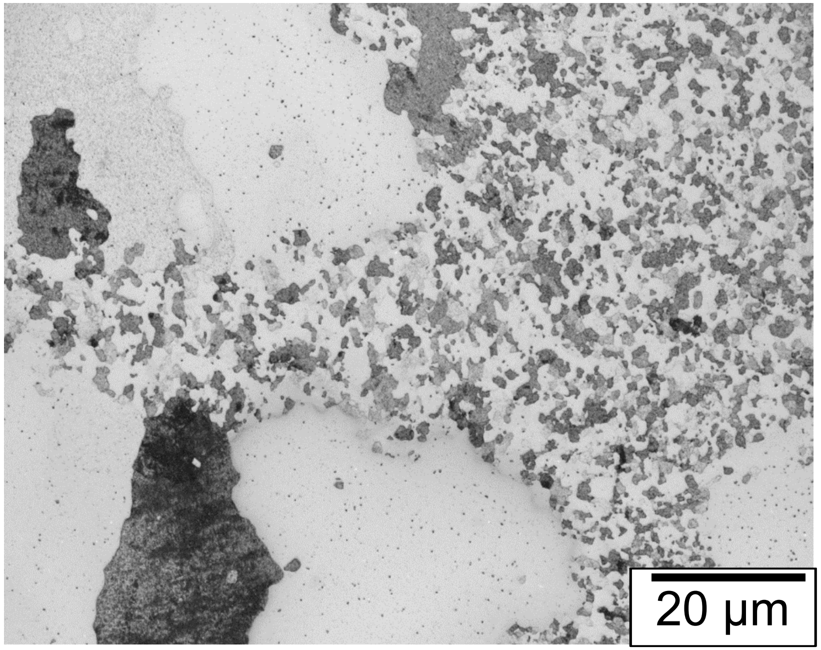
Unusual annealing behavior

- Applied 'standard' Hiperco anneal at 838°C (1111 K) for 2 hrs. in a high vacuum (<1E-5 Torr)





Highly heterogeneous recrystallization behavior – large grains consumed fine, as-built microstructure.

A unique bimodal grain structure

- Heterogeneous recrystallization also in cylinders.

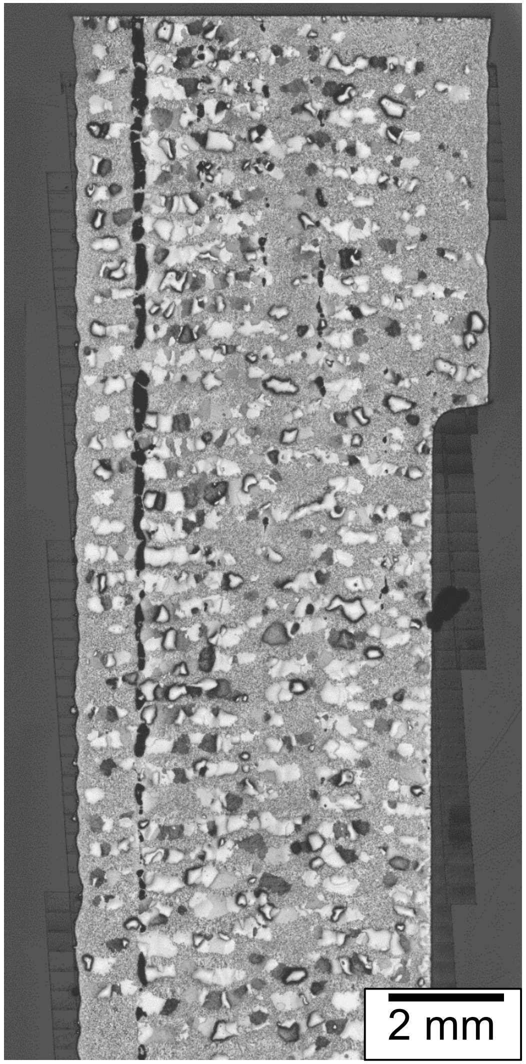
- Crystallographic texture was near-random.

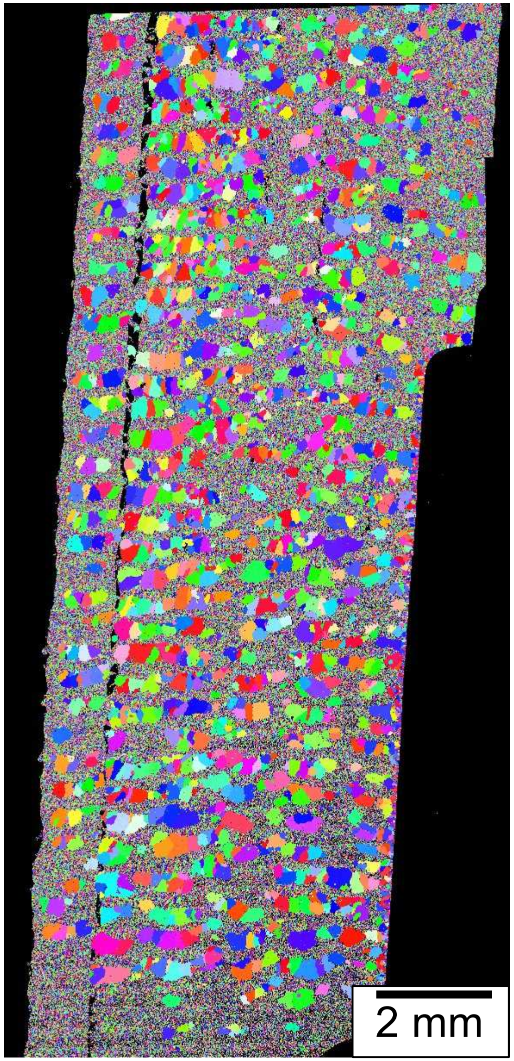
Why abnormal grain growth?

Selective grain boundary pinning processes promoted by:

1. Process-induced residual stress/strain pattern from LENS thermal history.

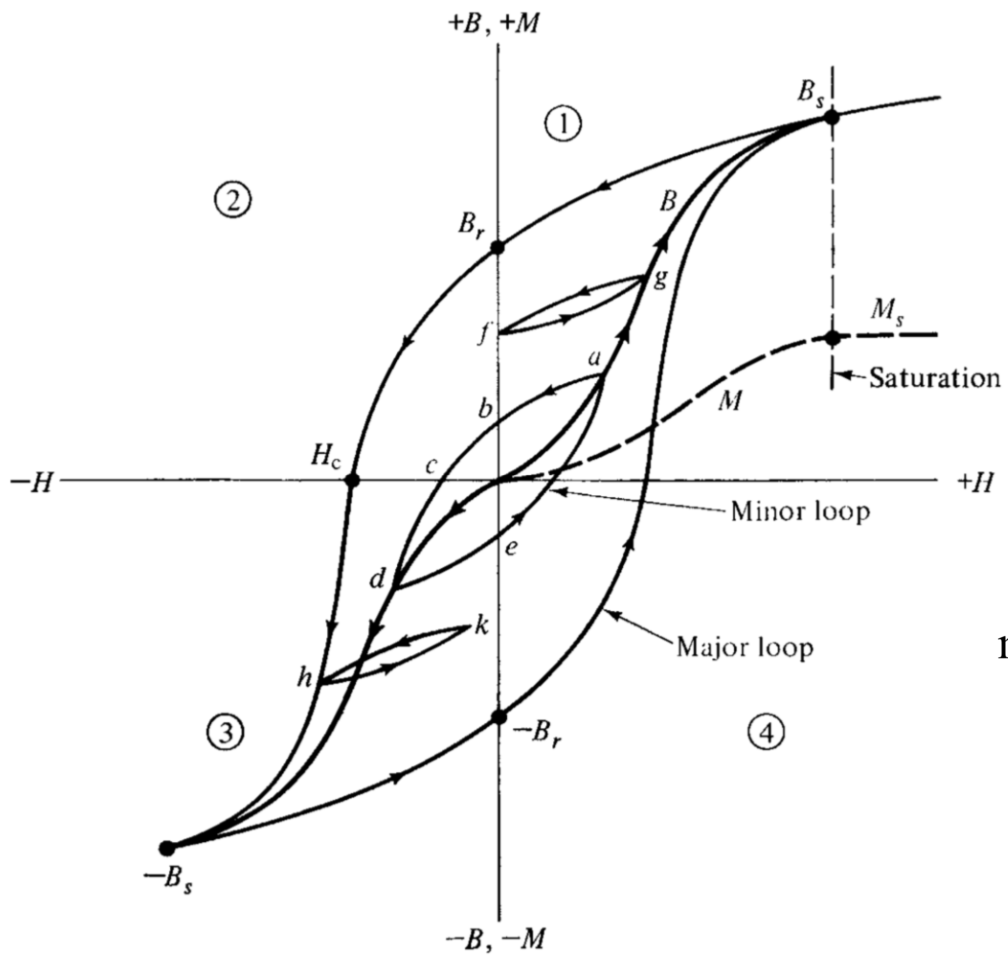
2. High density of precipitates that inhibit normal grain growth.





Magnetic Properties Characterization

Quasi-static Hysteresis Loop



<u>Key Properties:</u>

B_{max} – full-field induction

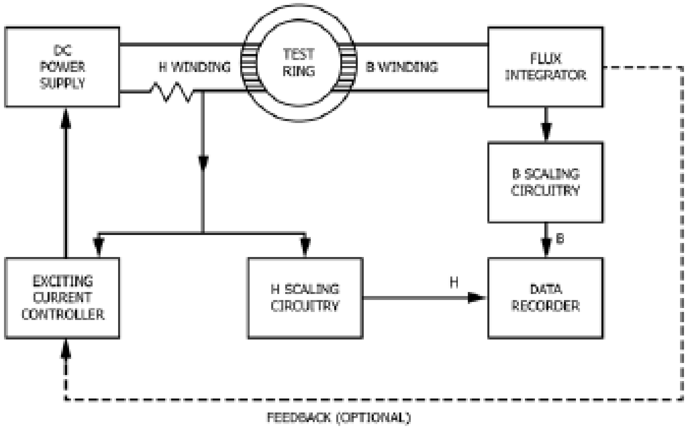
H_c – coercivity

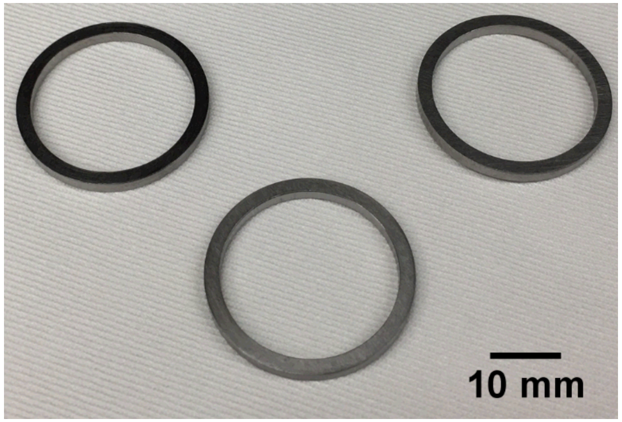
$\mu = B/H$ – permeability (slope of virgin magnetization curve) – here, focus on μ_{max}

Magnetic Properties Characterization

KJS Associates, Indianapolis, IN

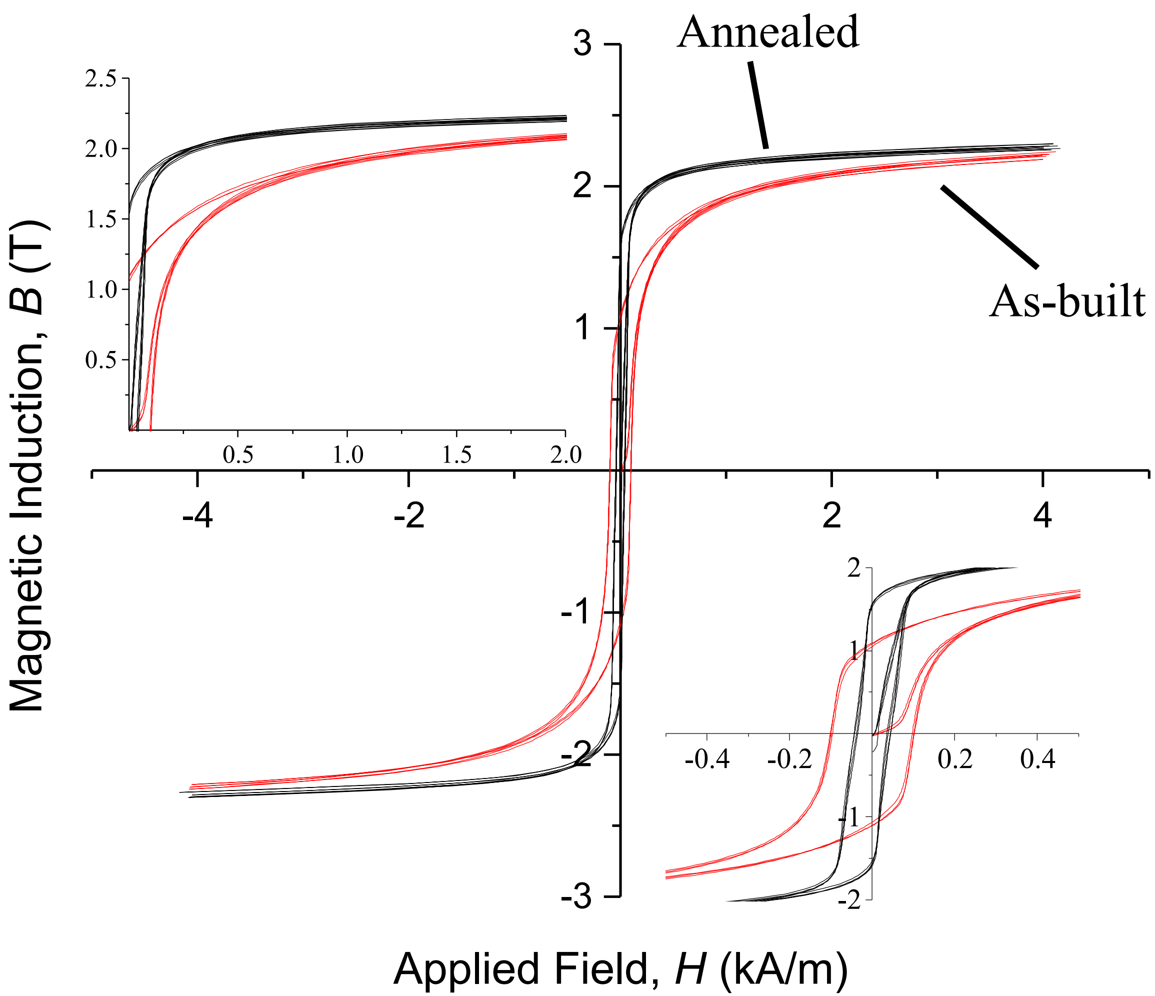
ASTM A773: Direct Current Magnetic Properties of Low Coercivity Magnetic Materials Using Hysteresigraphs





Fe-Co-1.5V B-H rings for magnetic properties characterization

Typical soft ferromagnetic behavior observed in specimens



As-built condition exhibited a more ‘sheared’ hysteresis loop – magnetically harder

Magnetic properties were within reported ranges

Goal is high permeability, low coercivity, and high full-field/saturation induction

- Annealed LENS condition: higher permeability and lower coercivity
- Values were within extremes of conventionally processed Fe-Co alloys.
- Tuning of post-processing annealing will continue to improve performance.

Condition	Specimen	Full-field Induction, B_{40}, (T)	Coercivity, H_c, (A/m)	Maximum Permeability, μ_m
As-built Fe-Co-1.5V	1	2.23	1013	511
	2	2.24	966	532
	3	2.21	1006	512
Average	--	2.23 +/- 0.5%	995 +/- 2%	518 +/- 2%
Annealed Fe-Co-1.5V	1	2.30	383	1639
	2	2.28	351	1733
	3	2.26	439	1571
	4	2.30	431	1517
Average	--	2.29 +/- 0.7%	401 +/- 9%	1615 +/- 5%
Fe-Co	--	2.4[25]*	150[25]	5000-8000[25]
Fe-Co-2V	--	2.3[25]*	90-200[62] 95-160[63] 393[25]	4000-8000[25]
Fe-Co-2V (as-rolled, 90%)	--	2.2[25]*	2900[25]	--

Conclusions

1. LENS was successfully demonstrated on Fe-Co and Fe-Si soft ferromagnetic alloys at conventional and close to ideal compositions.

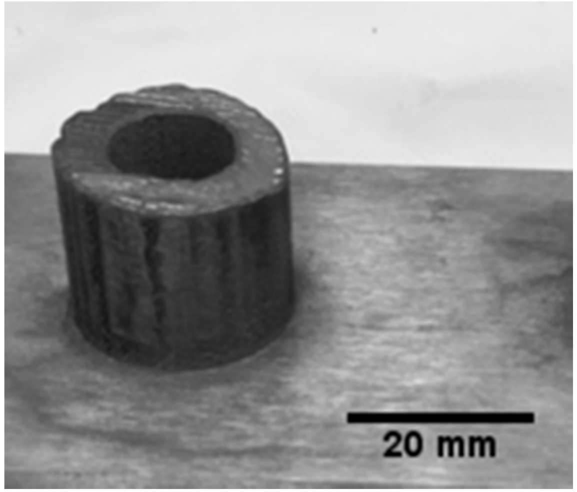
2. X-ray measurements on thin walls suggest reduced atomic ordering in as-built conditions, the degree of which was a function of cooling rate and thermal gradient sharpness.

3. Fe-Co specimens developed a fine equiaxed grain structure following layer-by-layer solidification, which evolved abnormally during annealing.

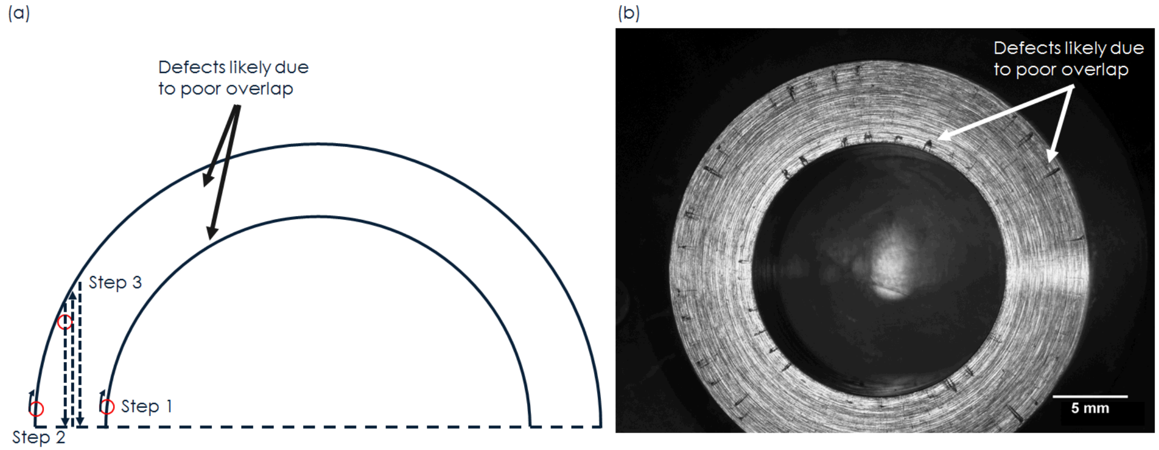
4. Magnetic properties of LENS Fe-Co competed with or exceeded conventionally processed Hiperco.

AM is an enabling process for low workability alloys at compositions that are difficult or impossible to process conventionally.

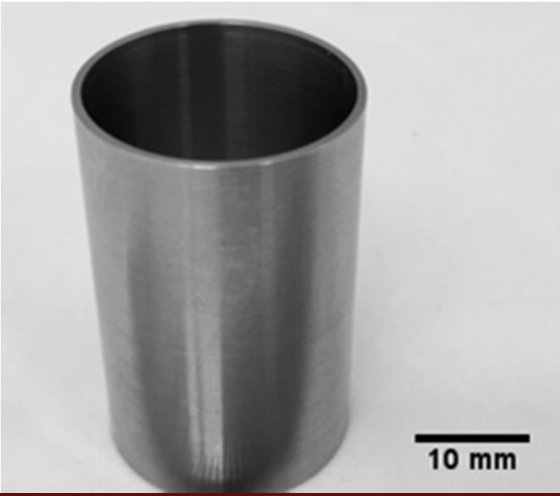
Great…. but build strategy matters!

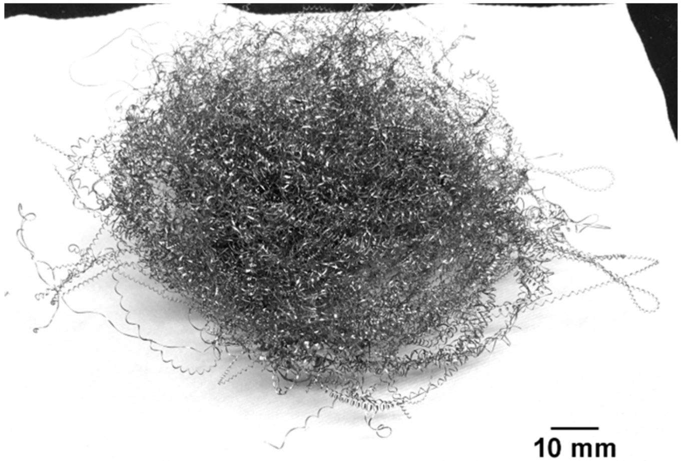


Defects from cross-hatch pattern.



BUT with a concentric build pattern, **no defects** and long continuous chips during machining, often a feature in cutting ductile metals (e.g., Al, Cu, Fe, etc.).

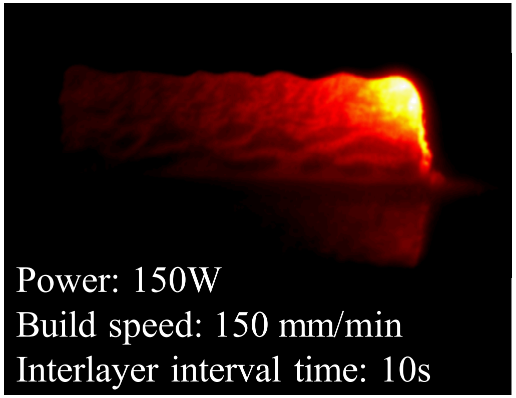


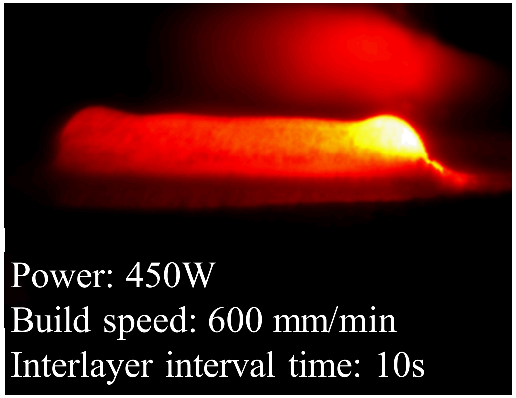


"…ductile metals tend to form long, continuous ribbon-shaped coils… such long continuous chips tend to form tangled nests…"

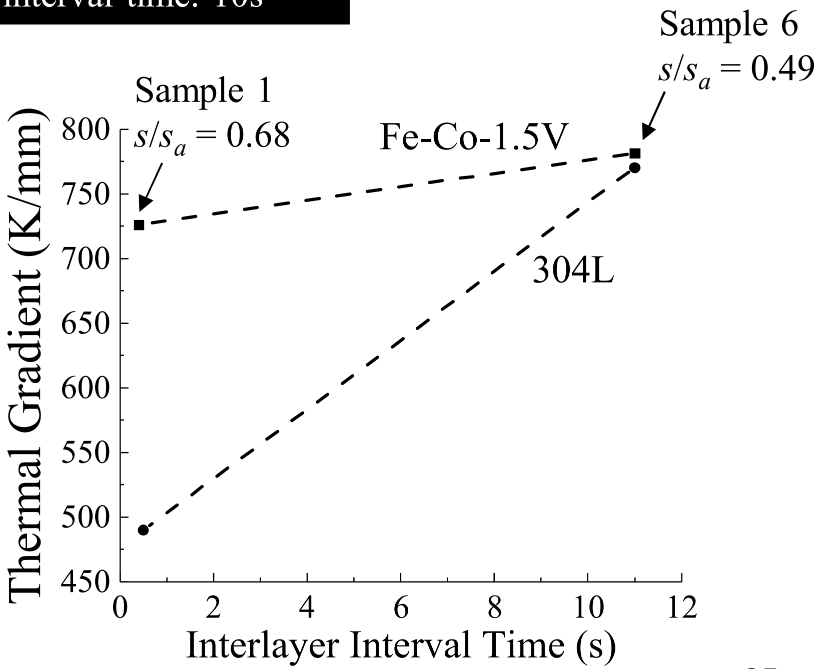
pg. 479 in 2nd edition of Metal Cutting Principles by Milton Shaw

Thermal measurements give some hints on the ordering results





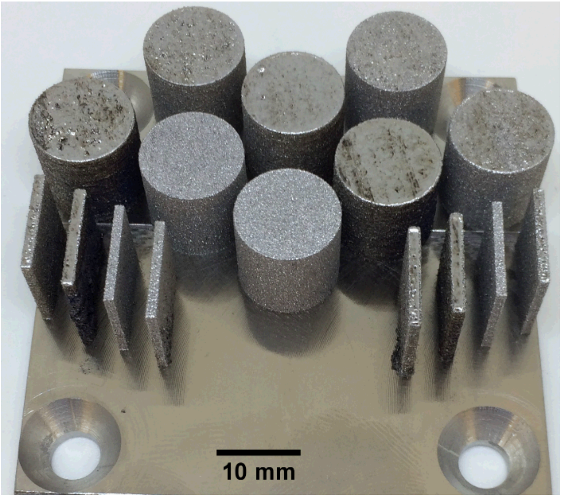
- Builds with faster interlayer interval time had sharper thermal gradients – less retained heat and less ordering.



Ongoing collaboration with Lehigh University (John Curry and Tomas Babuska (SNL), Prof. Brandon Krick)

Renishaw AM400 laser powder bed system

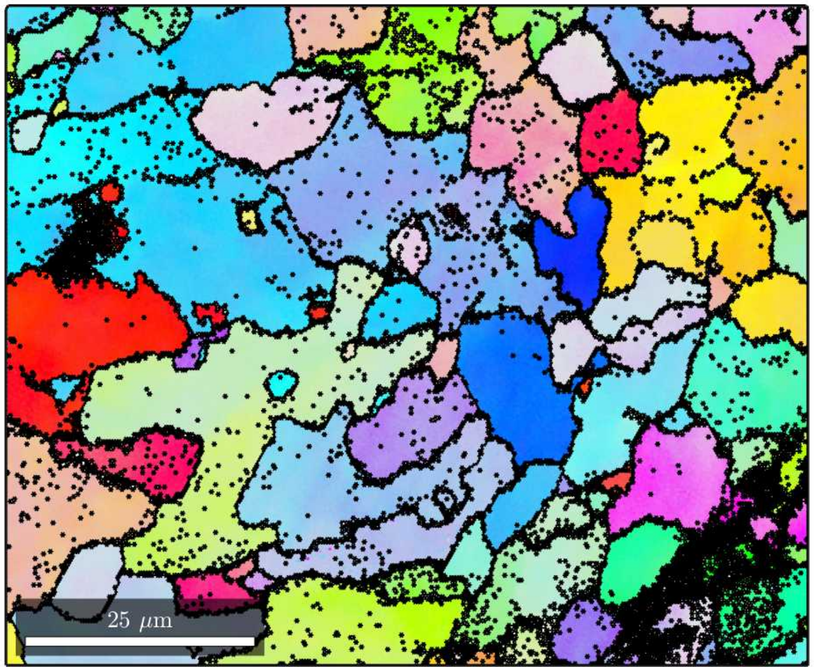
- Go/No-go again accomplished: successfully processed binary Fe-Co using powder bed.
  - Fe-Co too brittle for conventional processes!



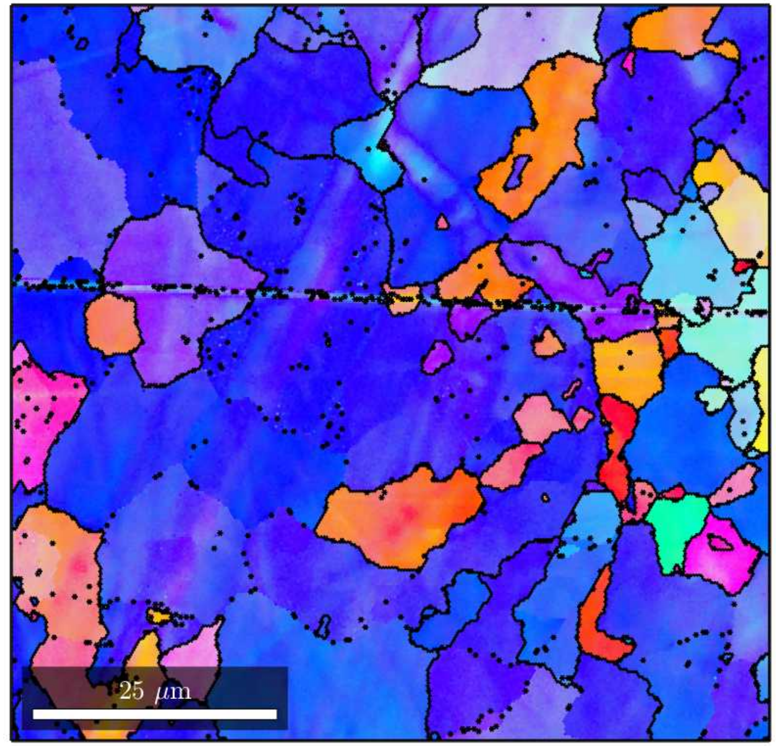
Lehigh University powder bed Fe-Co structures

Same fine equiaxed grain structure in powder bed samples

200W

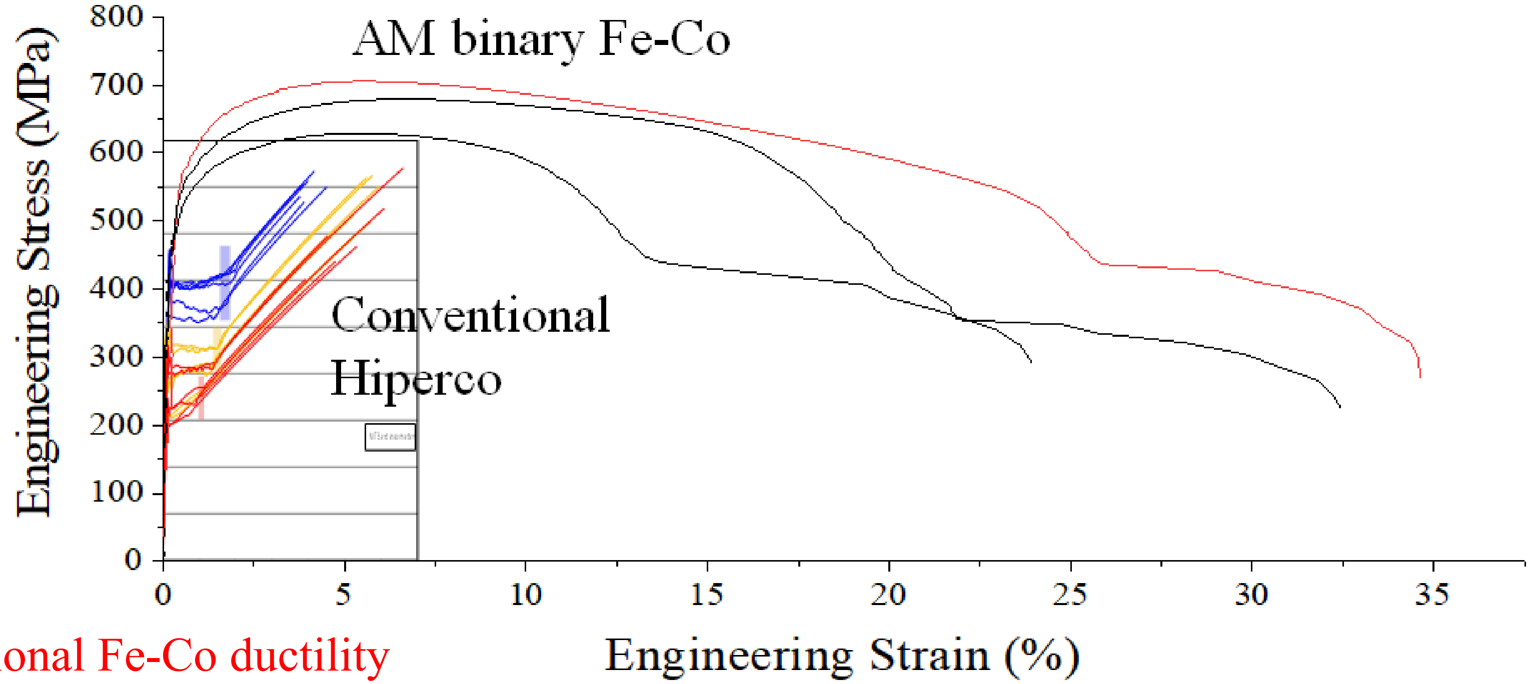


300W



- Fine, equiaxed grain morphology found in as-built AM powder bed specimens.
- Currently assessing the crystallographic texture.

Excellent mechanical properties for binary Fe-Co



Conventional Fe-Co ductility

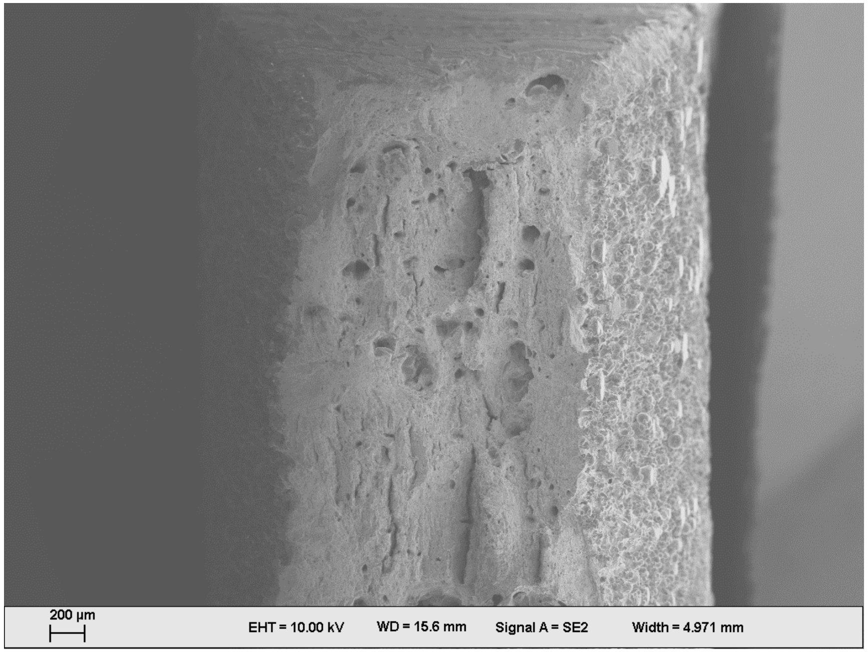
Alloy	Condition	Elongation
Fe–50Co	Ordered (2 h at 800 °C + 10 h at 550 °C)	4%
Fe–50Co	Disordered (2 h at 800 °C + IBQ)	0%
Fe–49.3Co	Ordered (heat-treatment details unknown)	1.6%
Fe–49.3Co	Disordered (as above)	0%

Sourmail, 2005, Prog. Mater. Sci.

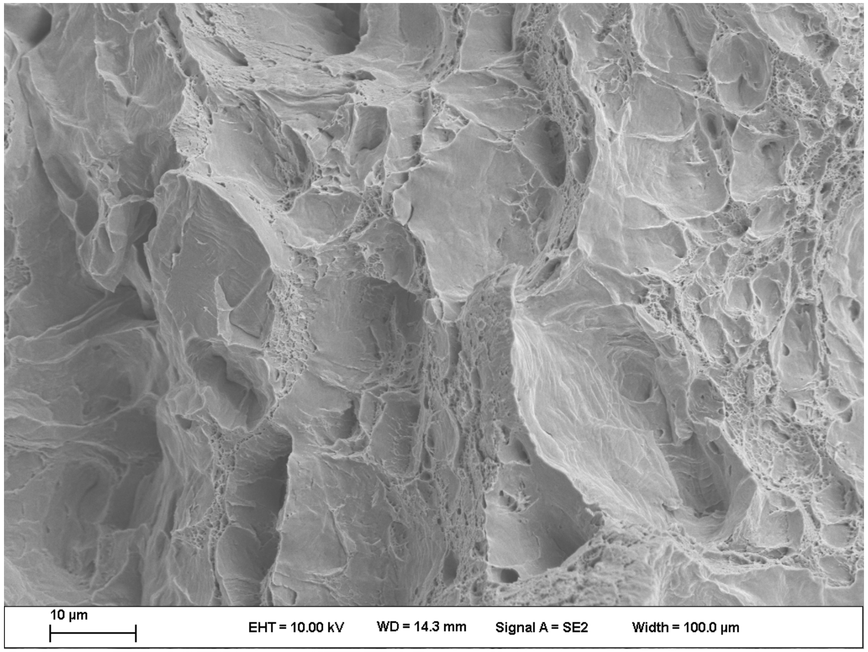
Higher strength and ductility for AM binary Fe-Co.

World record ductility?

Extensive necking and microscale plastic flow!!



Extensive necking and micro-void formation



Microscale plastic flow on the fracture surface

New deformation attributes for Hiperco/Fe-Co!