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Laser Additive Manufacturing of Fe-Si Based Soft Magnetic Alloys

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Sandia National Laboratories

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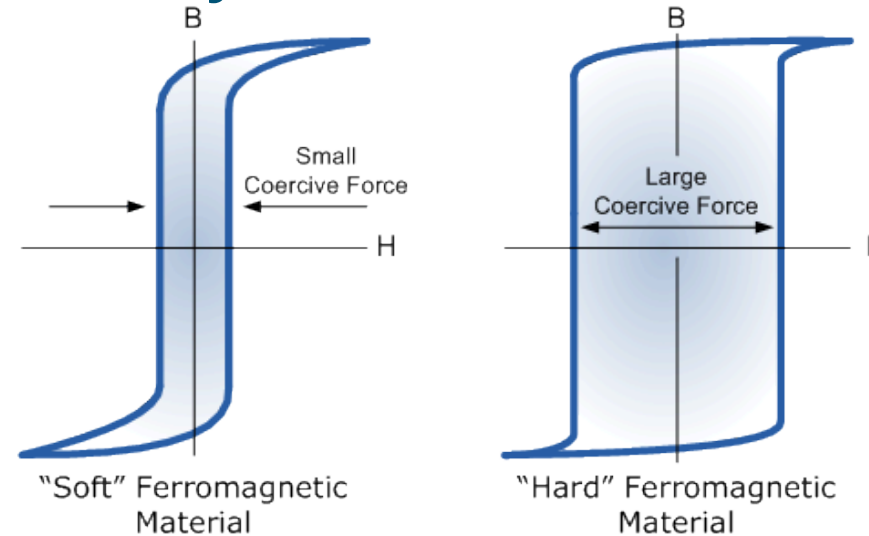




Fundamentals of soft magnetic alloys

Excellent soft magnetic properties:

- High saturation induction
- High permeability (High B for low H)
- Low coercivity (narrow loops)
- Low core loss (narrow loops)
- Electric motors, transformers, switches, etc.

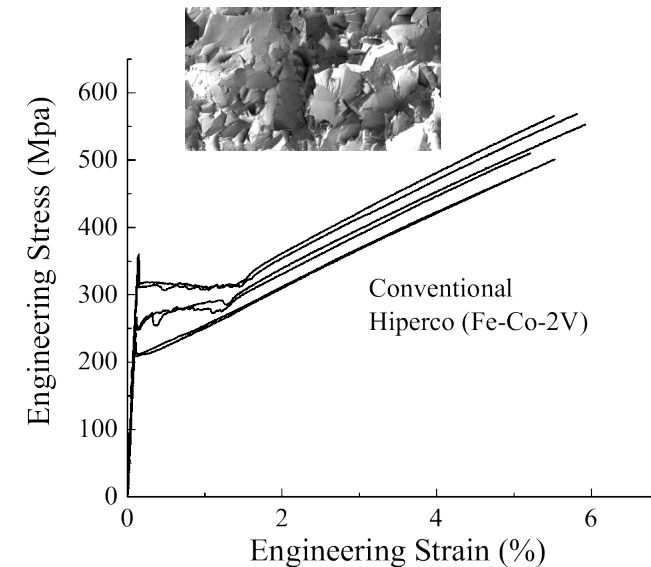
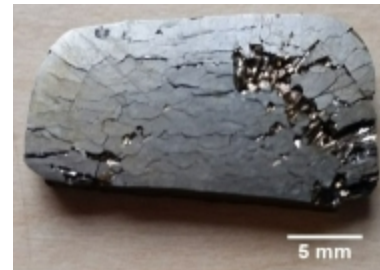


- However -

Poor mechanical properties:

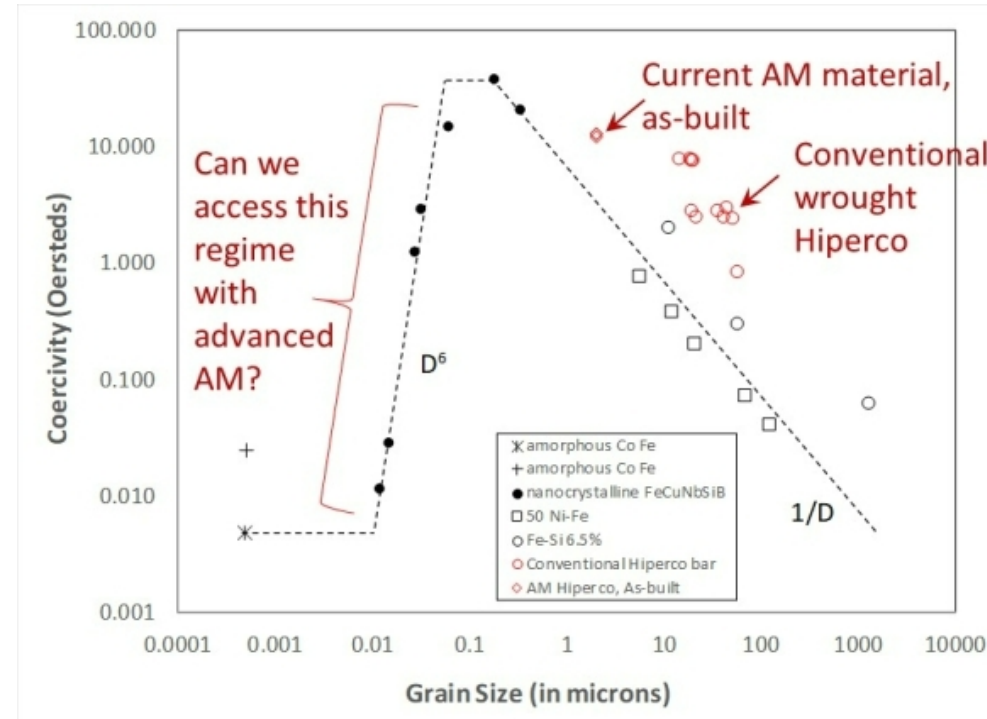
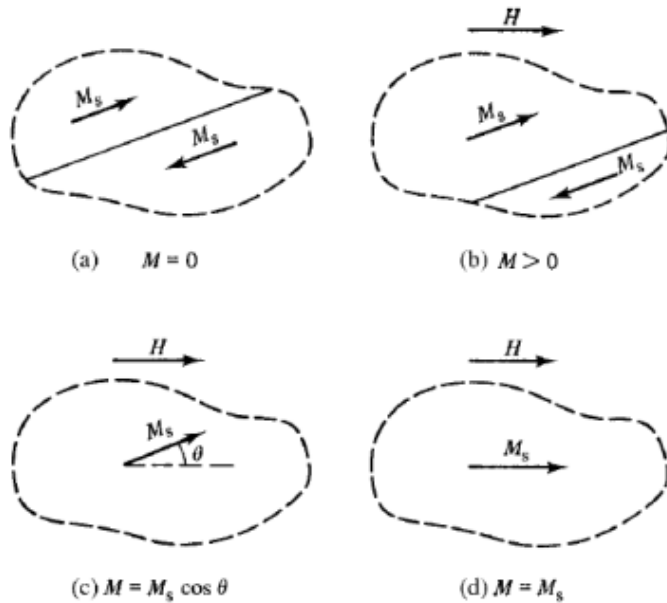
- Result of ordered B2 and DO3 phase transformations
- Low yield strength
- Low ductility
- High notch sensitivity
- Low fracture toughness
- Low fatigue resistance

High silicon content electrical steel (Fe-6.5wt%Si)





Why these property tradeoffs?



Fundamental challenge: structural barriers (i.e., line and area defects, etc.) increase strength/ductility but inhibit microscale magnetization processes.

Grain size has strong effects:

If $D = 0.1$ -1 micron, higher strength/ductility, harder magnetic properties.

If $0.1 > D > 1000$ micron, decreasing strength/ductility, softer magnetic properties.

Conventional processing challenging/impractical to achieve $D < 0.1$ micron in bulk.



AM processing of advanced soft magnetic alloys – building on previous literature

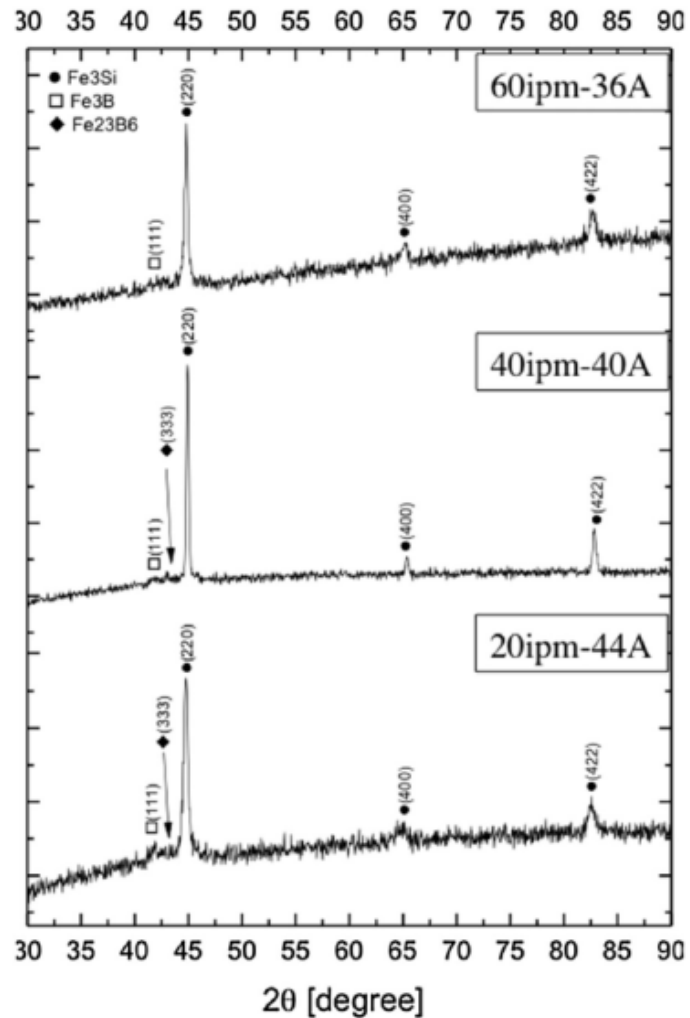
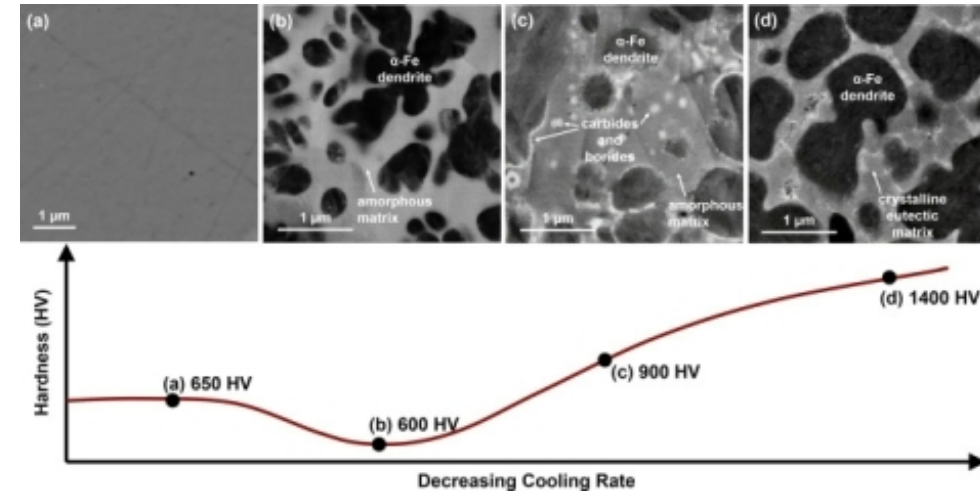


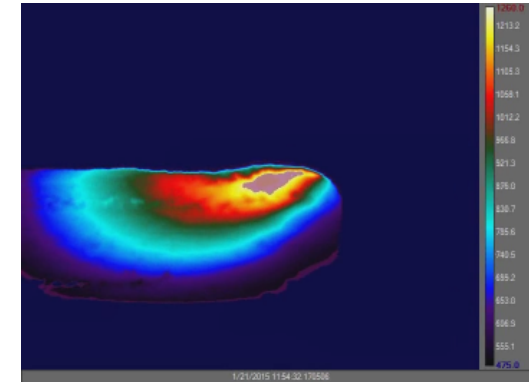
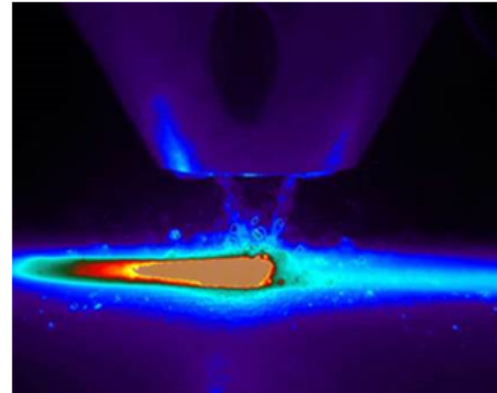
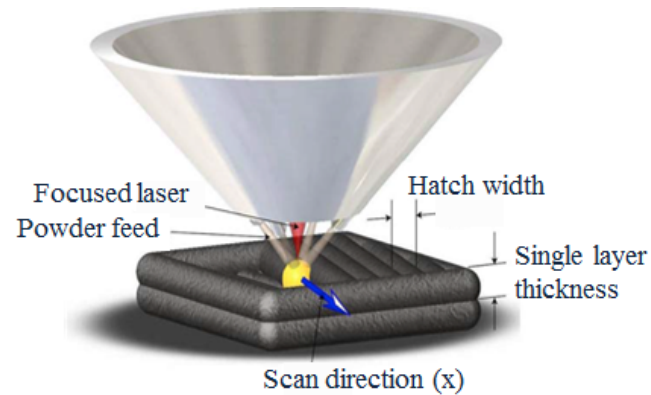
Fig. 2. XRD patterns of LENS™ processed FINEMET alloys.

Conteri et al. (2017) "Laser additive processing of Fe-Si-B-Cu-Nb magnetic alloys," Journal of Manufacturing Processes, Vol 29, 175 – 181.

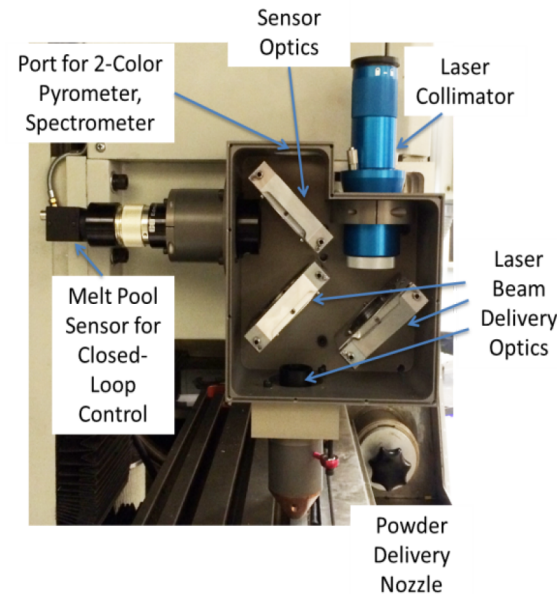
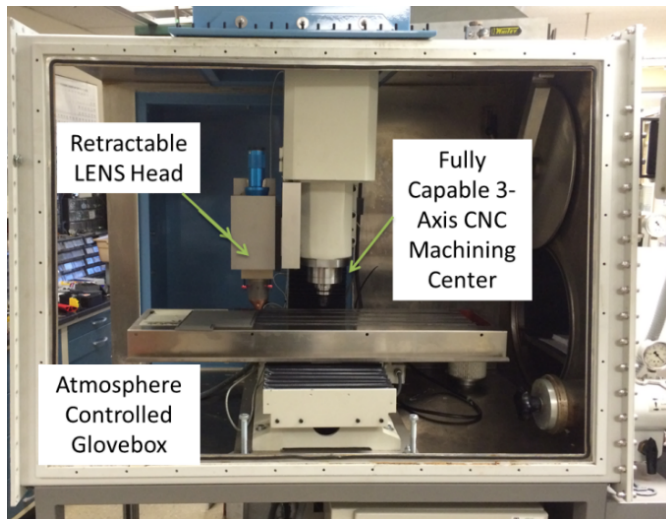


Bordeenithikasem, et al. (2021) "Controlling microstructure of FeCrMoBC amorphous metal matrix composites via laser directed energy deposition", Journal of Alloys and Compounds, Vol. 857, 157537

Laser beam directed energy deposition



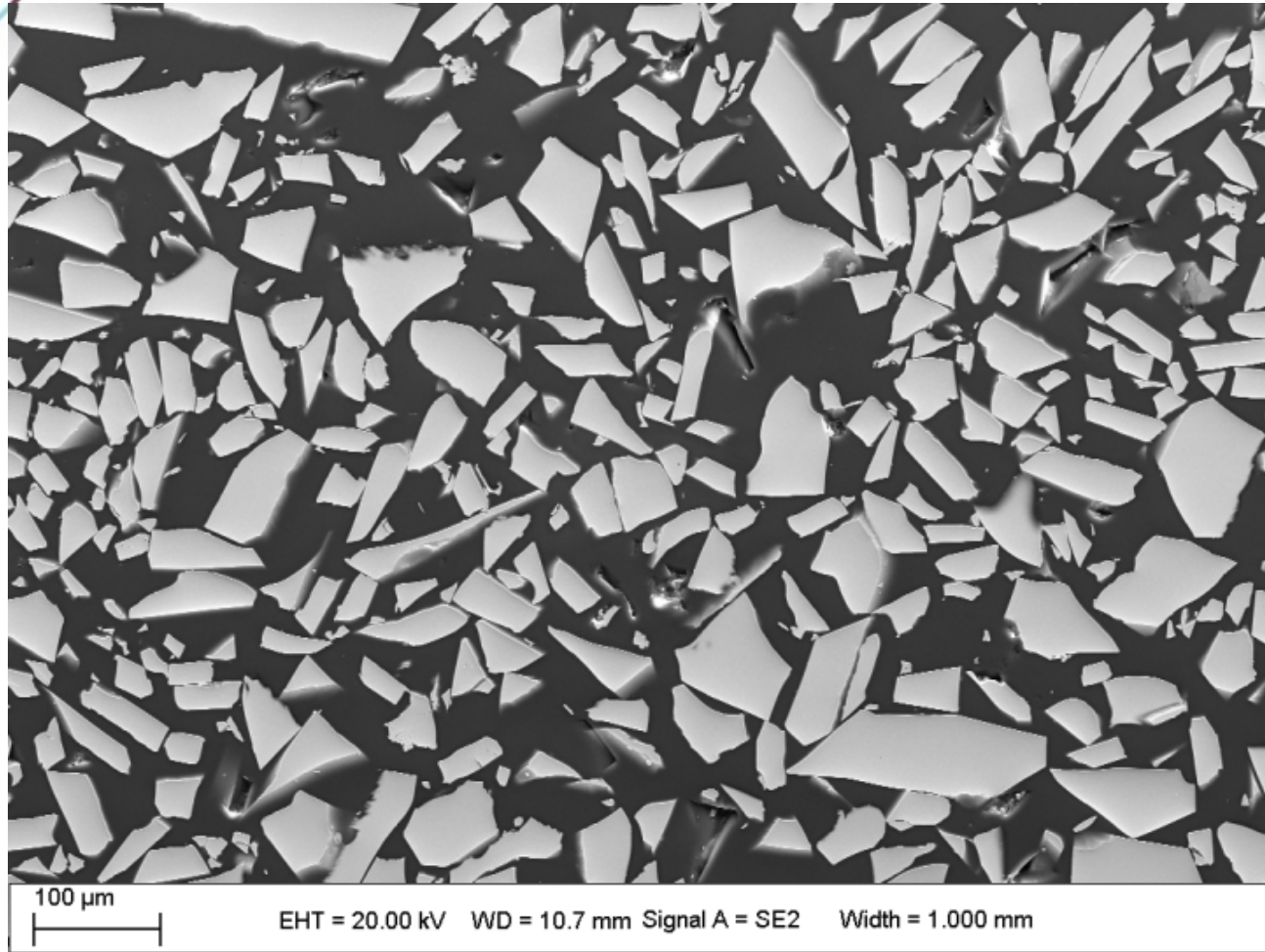
thermal history during bi-directional metal deposition



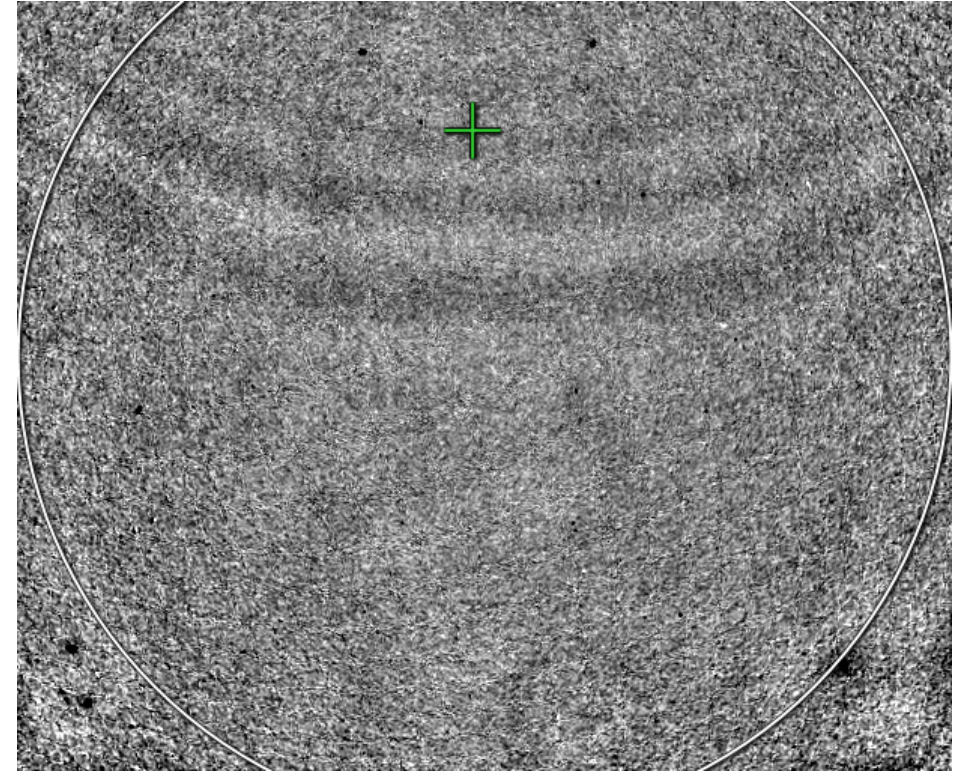
- Open architecture Laser Beam Directed Energy Deposition (LB-DED) apparatus for multi-material and custom alloy printing.
- 2-color pyrometer and FLIR cameras for in situ melt pool geometry and temperature measurements.
- High temperature heating platform for in situ stress-relief/annealing.
- Hybrid AM and subtractive processing.
- Controlled powder feed rate with up to 5 independent powder chemistries – enable in situ alloy design studies.



Powder feedstock – ball milled (amorphous) flakes



Provided by co-author Dr. Eric Theisen at Metglas



Target Alloy Composition:

- Fe = 83 wt %
- Si = 8.6 wt %
- Nb = 5.6 wt %
- B = 1.5 wt %
- Cu = 1.3 wt %



AM processing parameters

Produced thin-wall geometries

High relative density (>99%)

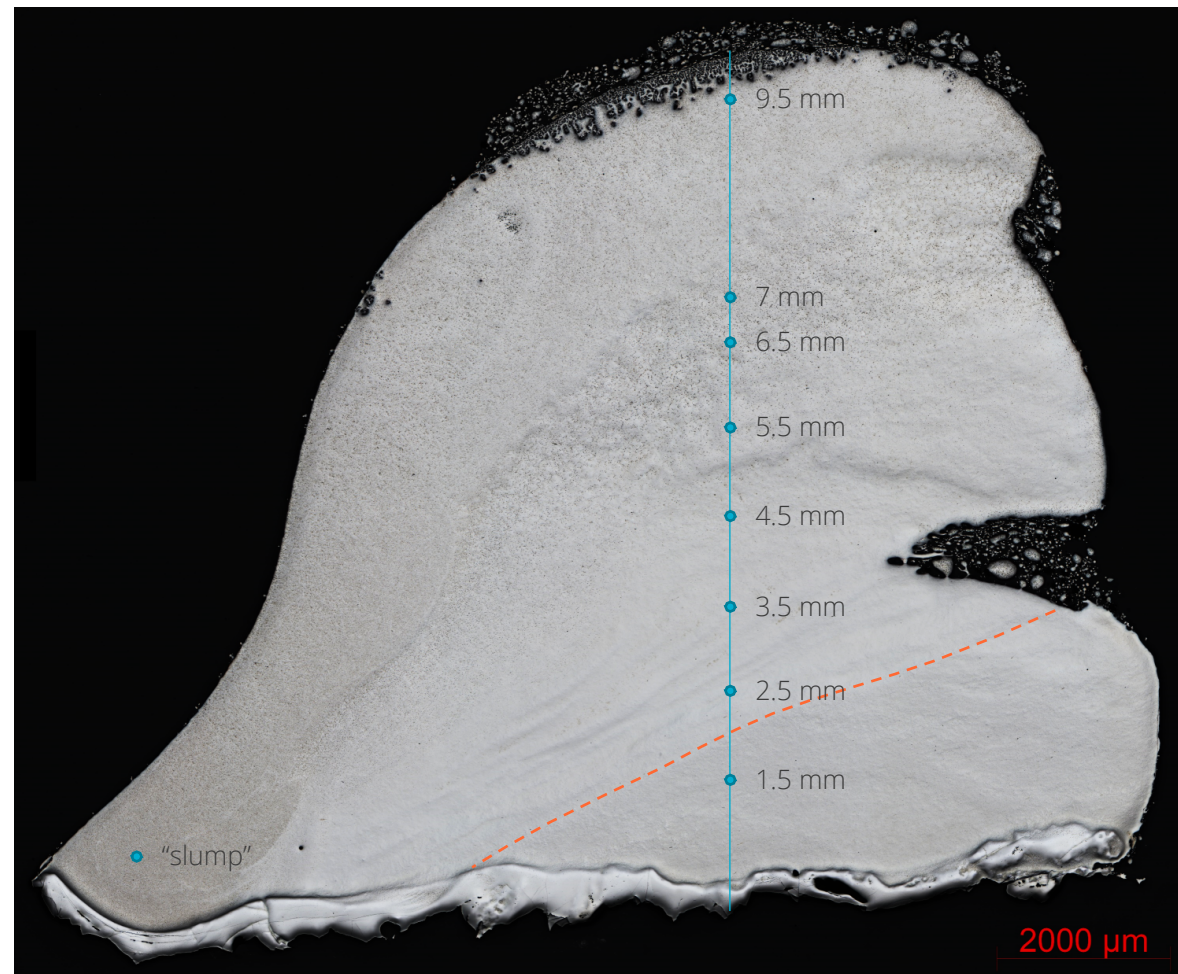
Laser Power: 400 W

Build Velocity: 450 mm/min

Substrate: Ti grade 2 (commercially pure Ti)

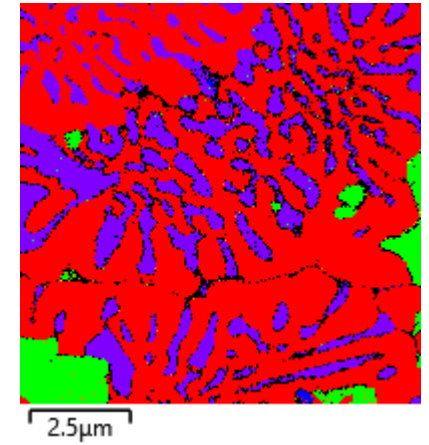
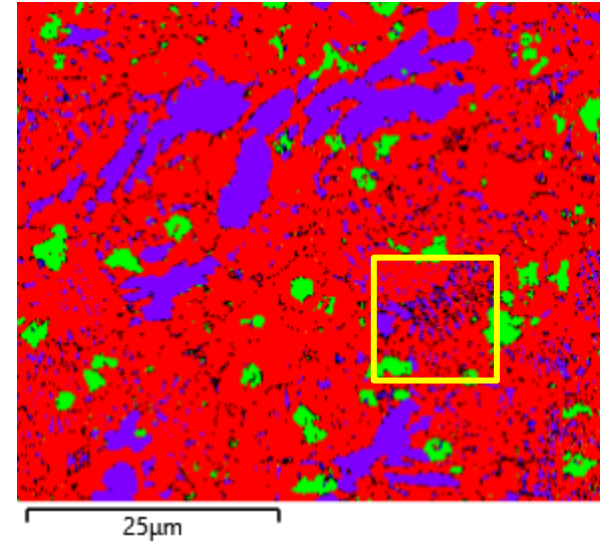
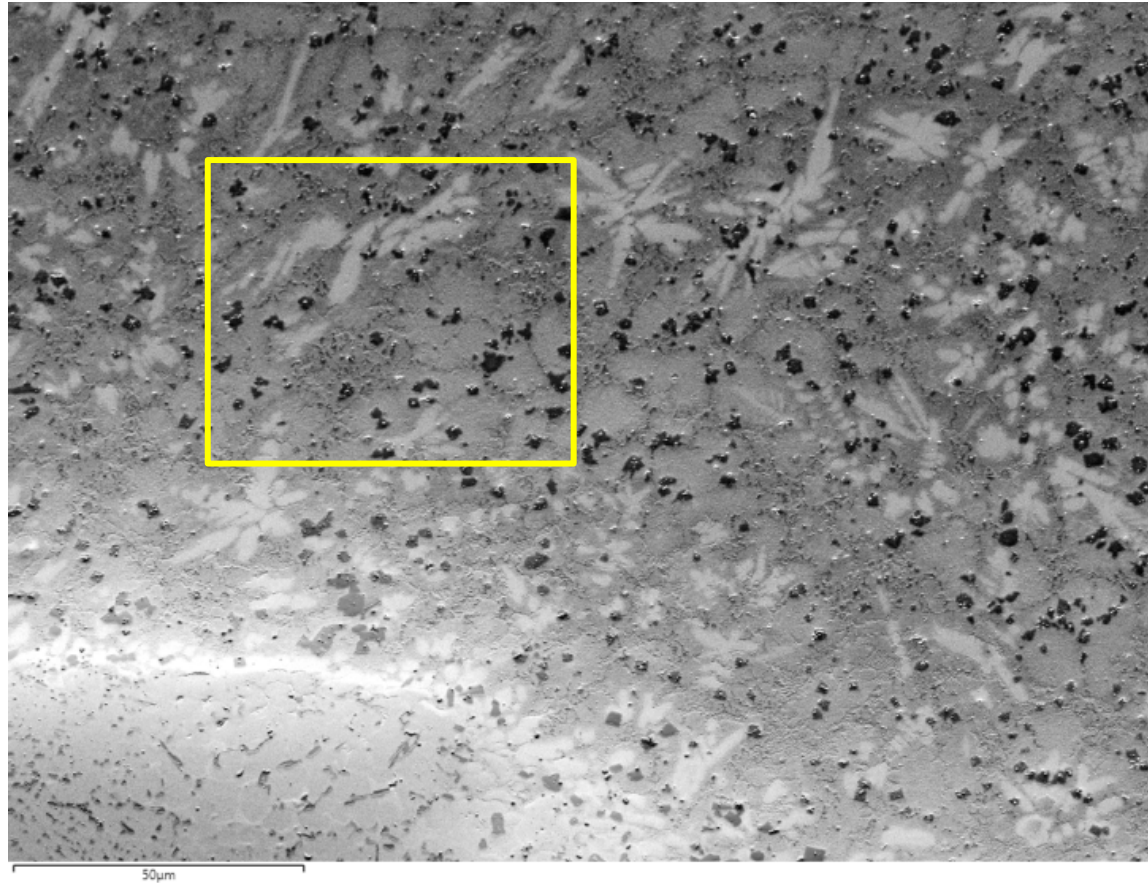
Was a nanocrystalline structure created??

Regions for SEM analysis (named for distance from bottom)

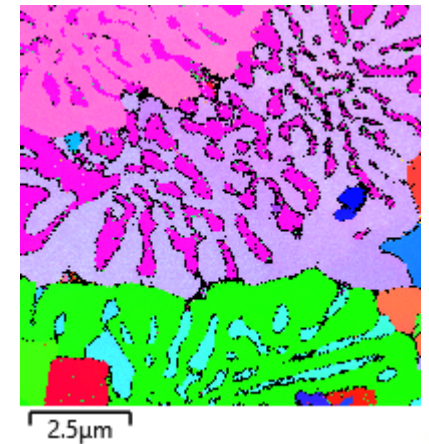
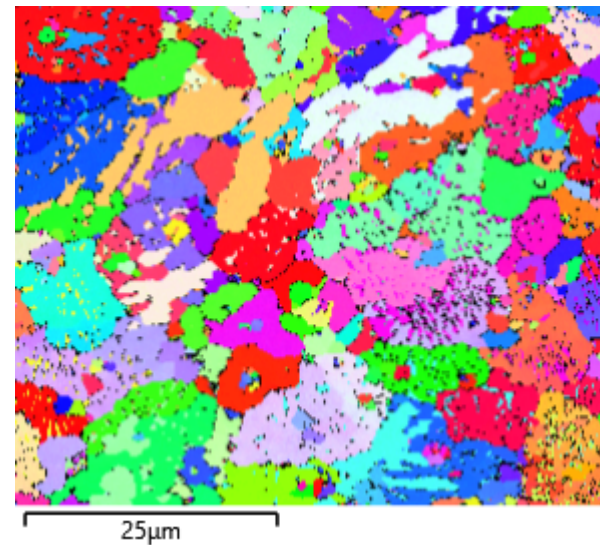




Near-bottom EBSD

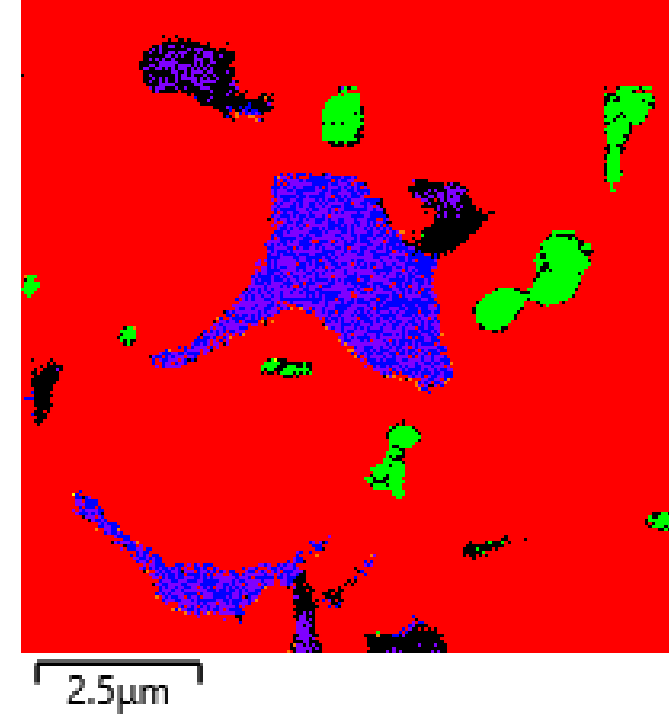
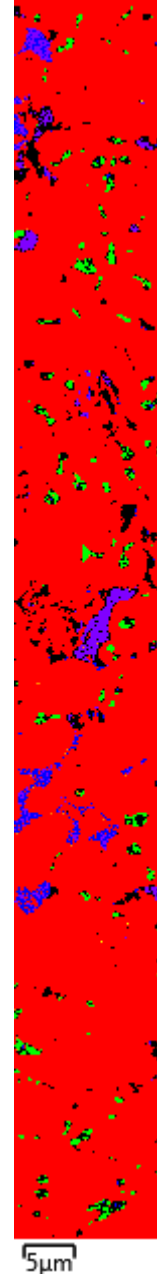
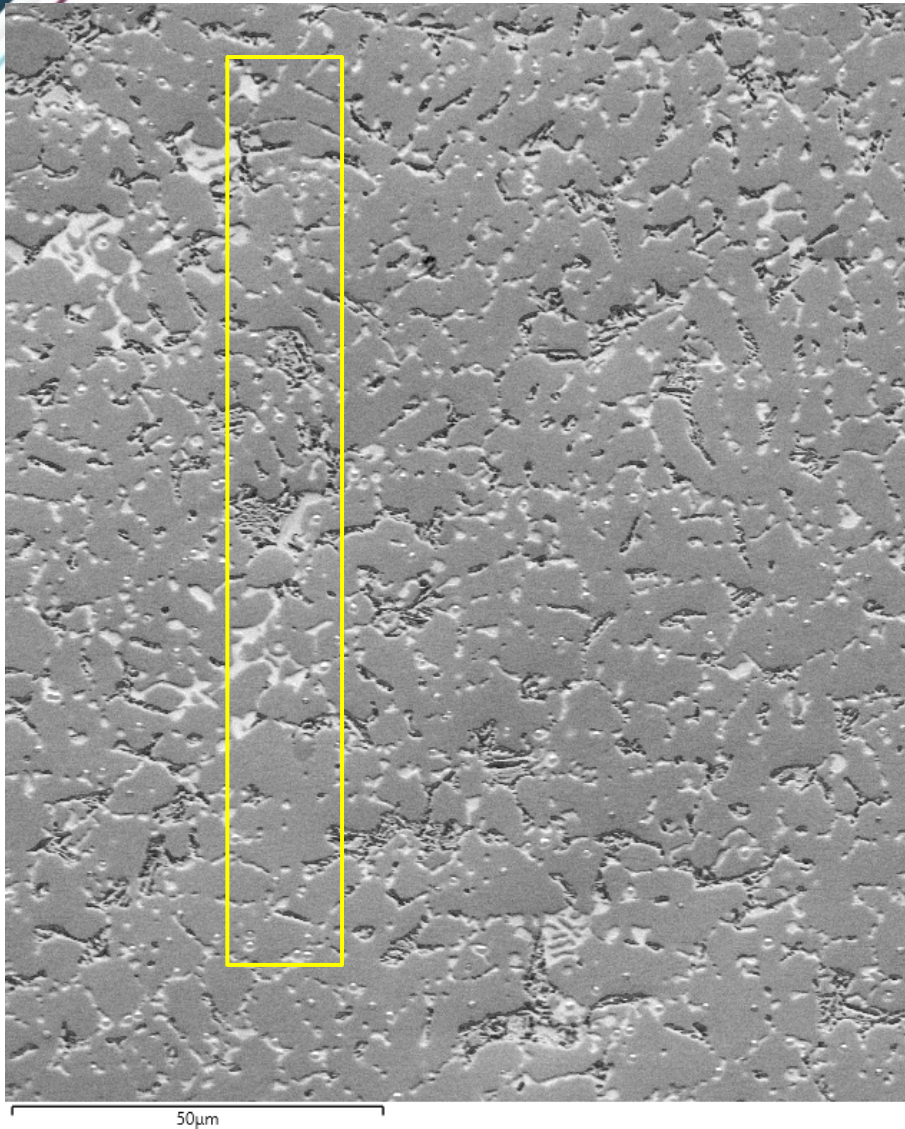


- BCC matrix
- Hexagonal NbFe₂ (Laves)
- Hexagonal B, Ti, Nb (could be TiNbB₄, TiB₂, or NbB₂)





Near-top EBSD



- BCC matrix
- Hexagonal NbFe₂ (Laves)
- Hexagonal B, Ti, Nb (could be TiNbB₄, TiB₂, or NbB₂)

Was a nanocrystalline structure created??

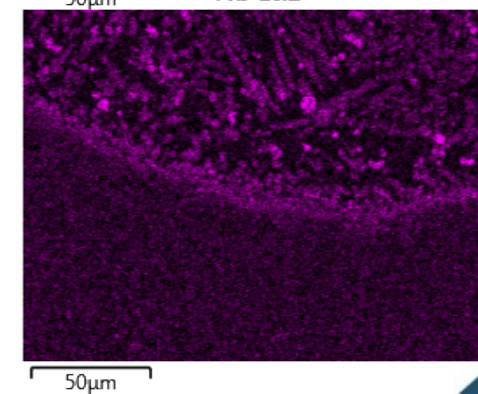
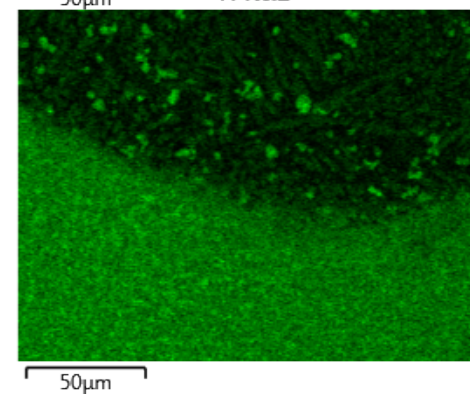
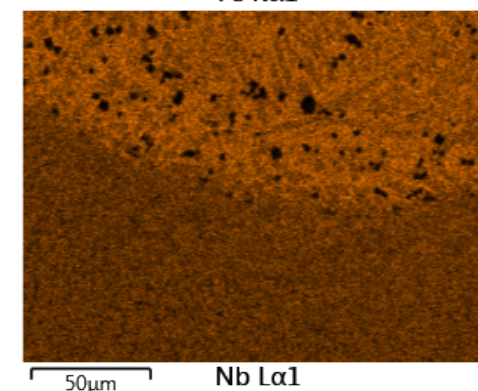
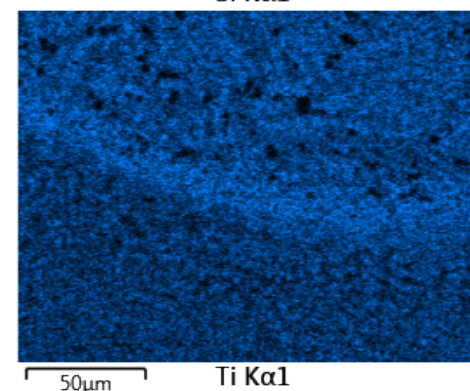
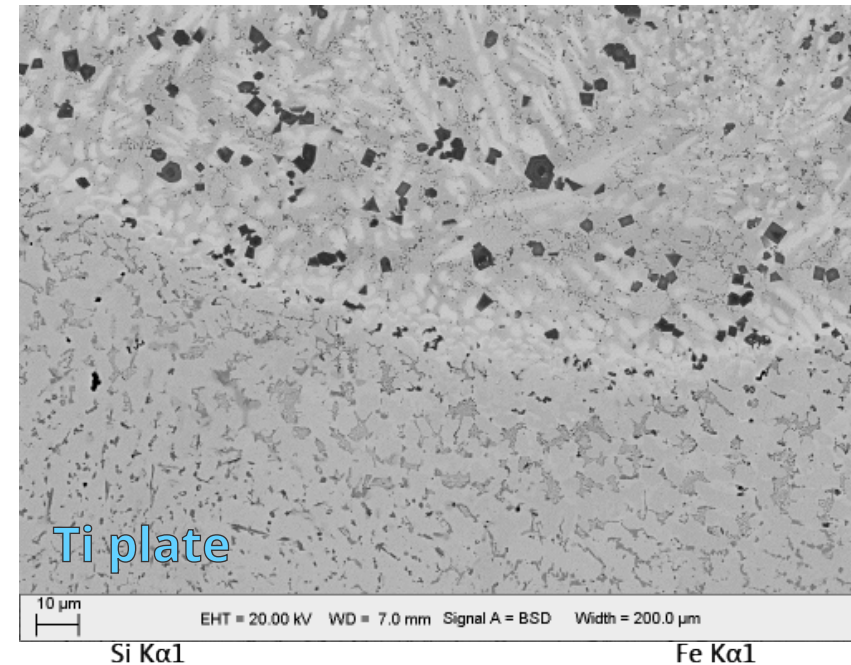
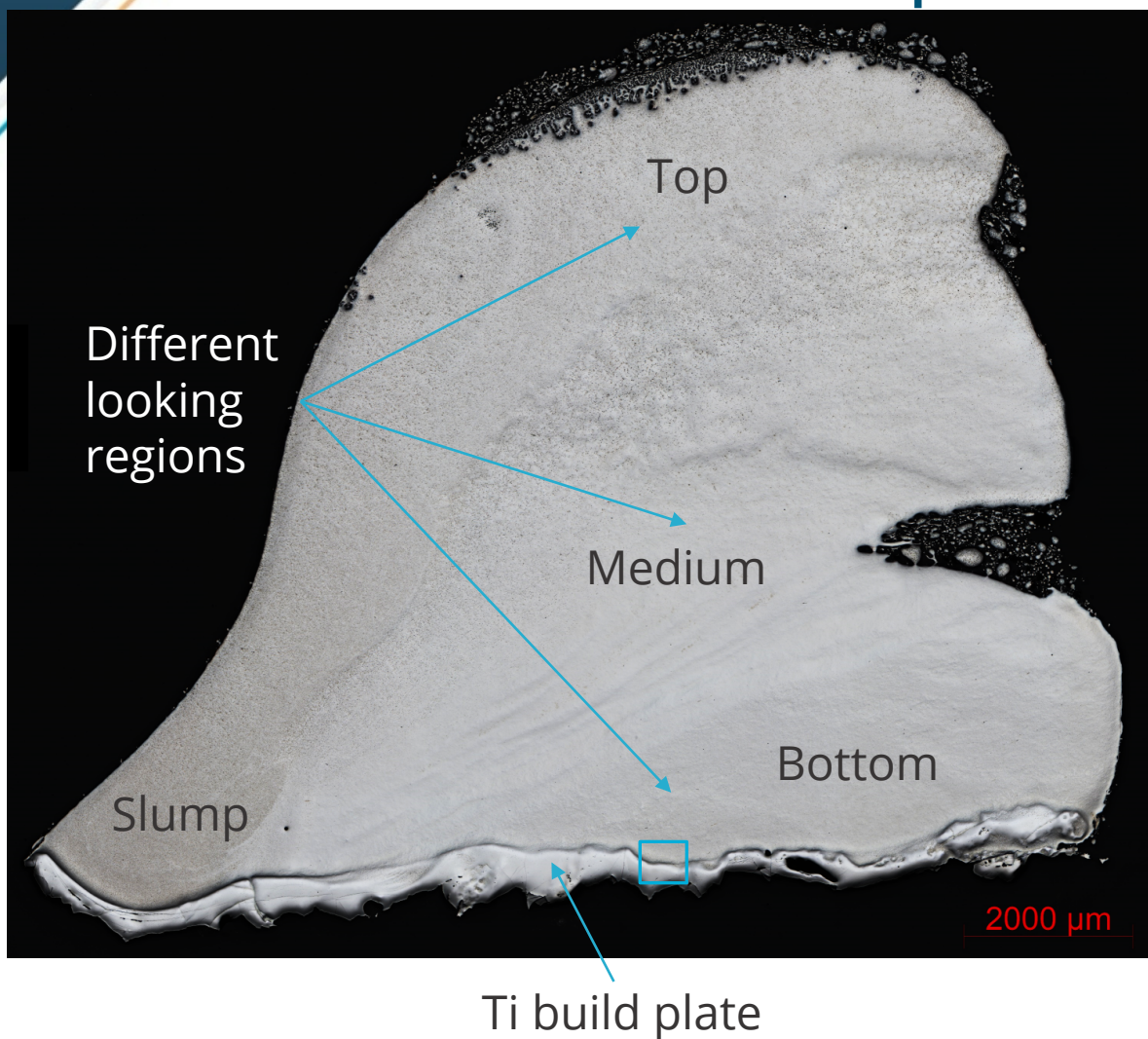
Coarse microstructure following DED.

Extent of bcc matrix phase increased with build height.

Characterization provides clues to grain structure...

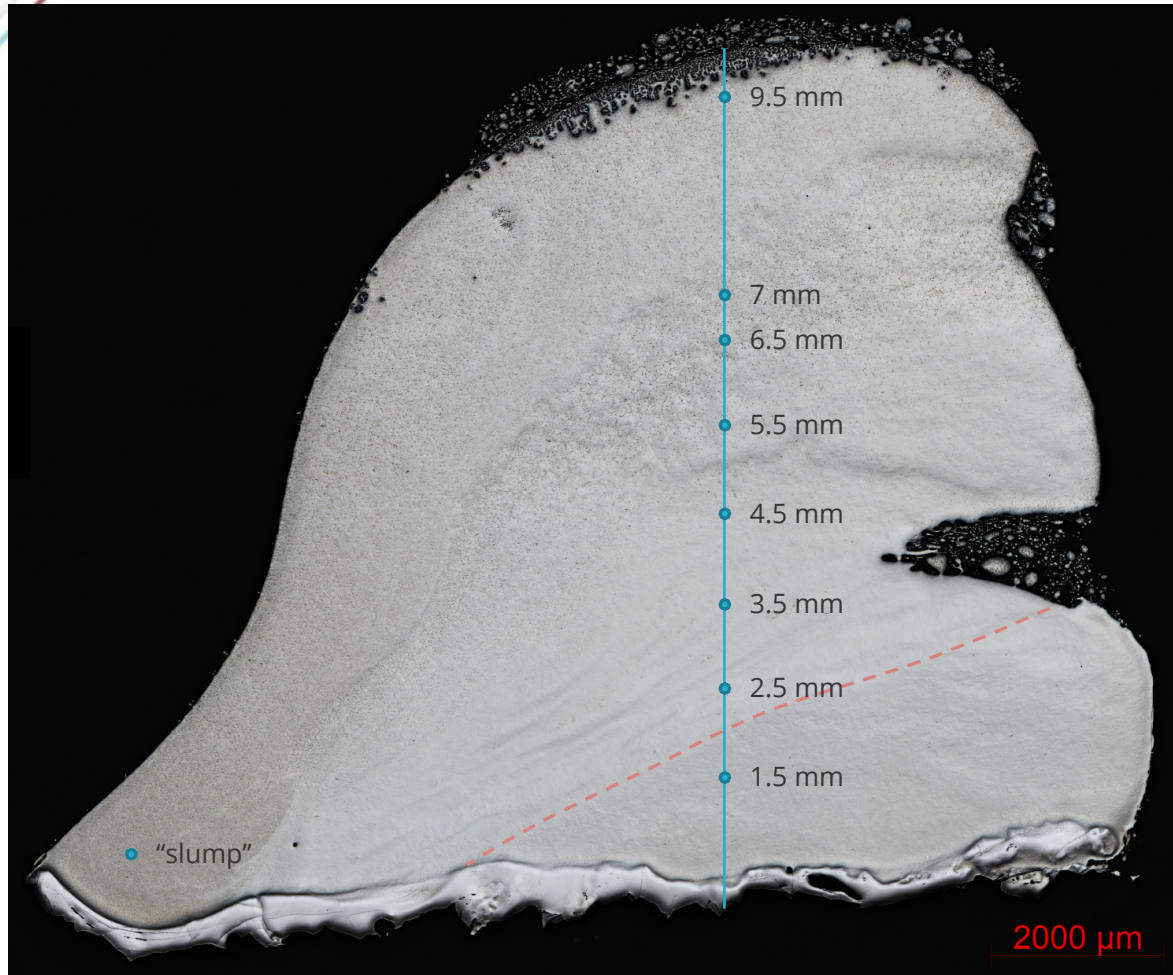


Interface with build plate

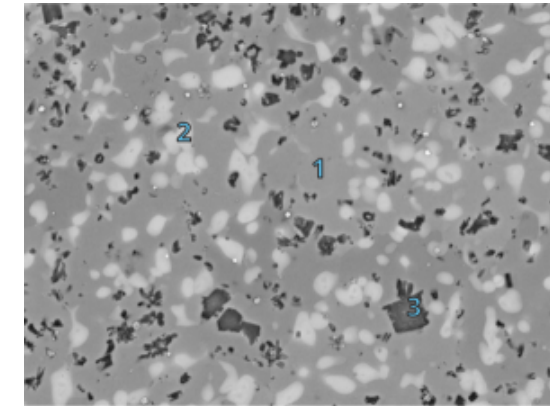




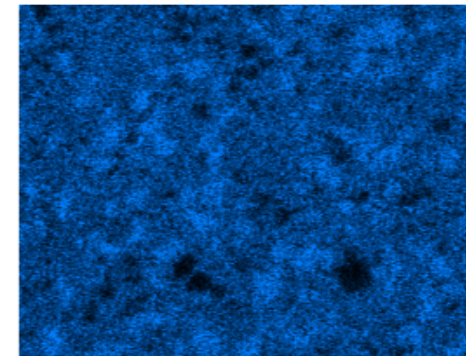
Bottom region – high Ti-content



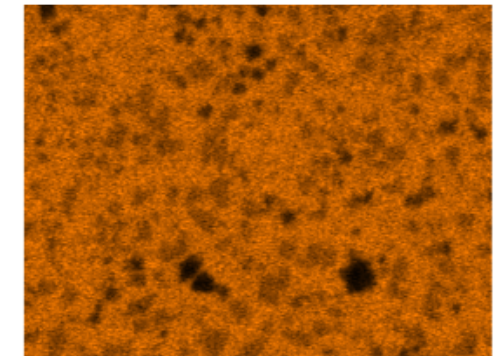
- 1: Matrix = Si & Fe
- 2: Bright Constituent = Nb, Si, Fe, & Ti (Fe in highest proportion)
- 3: Dark Constituent = Ti & Nb + **B** (B in highest proportion)



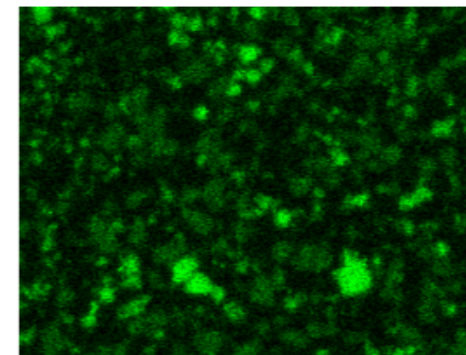
Si K α 1



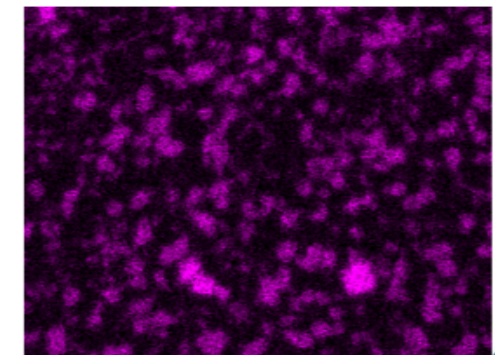
Fe K α 1



Ti K α 1



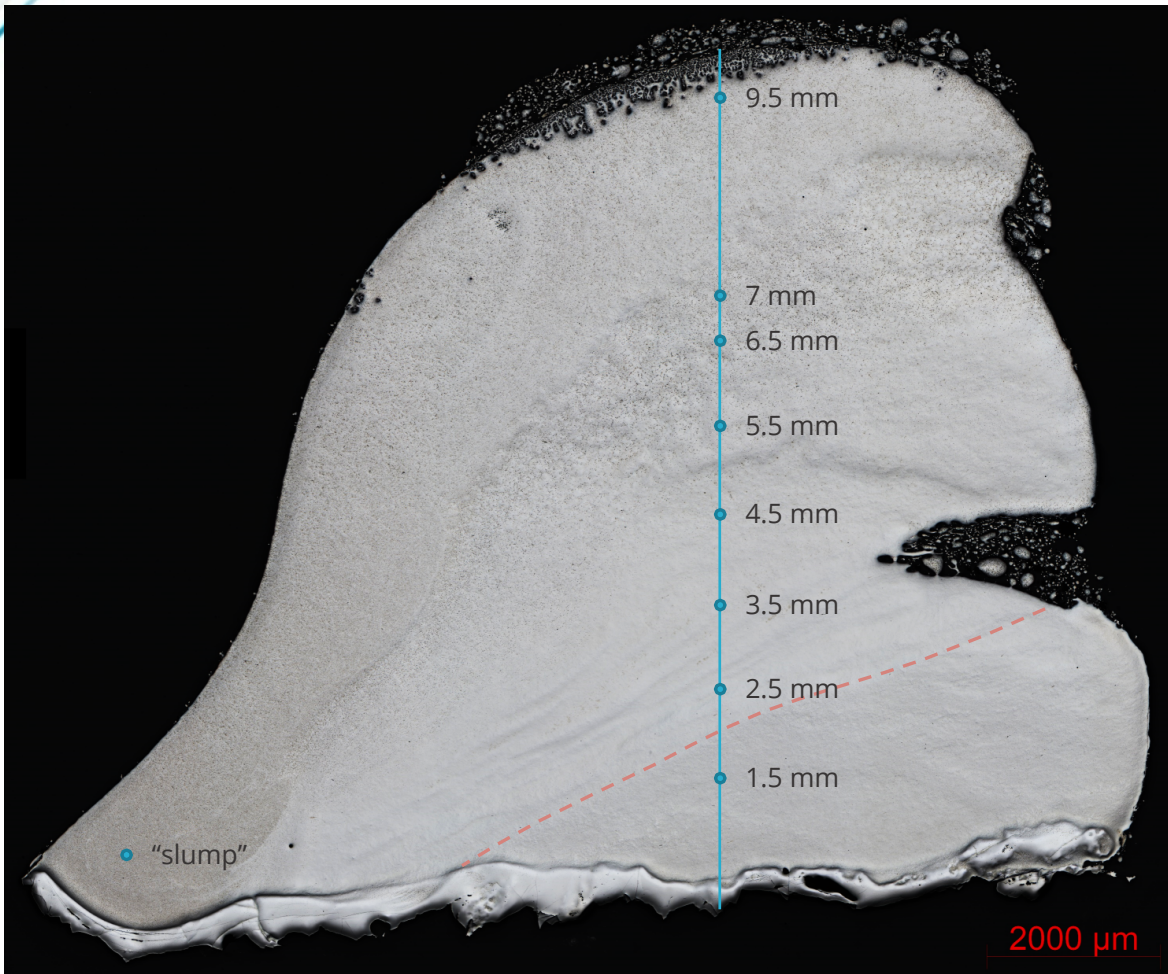
Nb L α 1



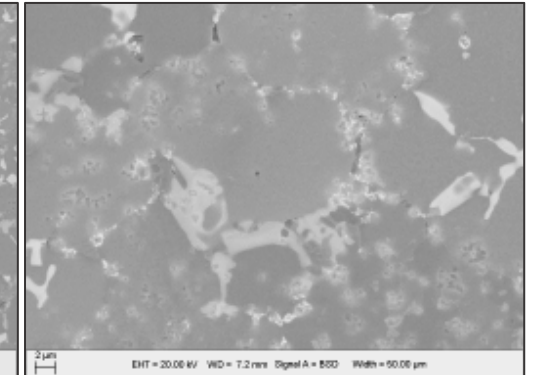
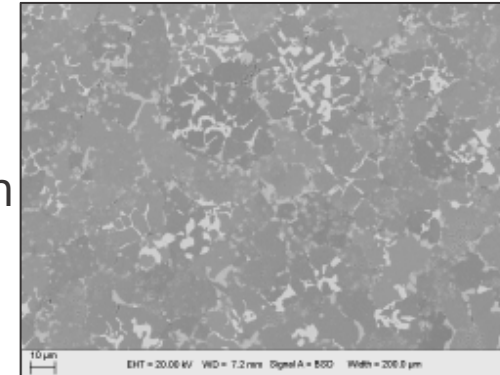


Middle region: 2.5-5.5 mm build height

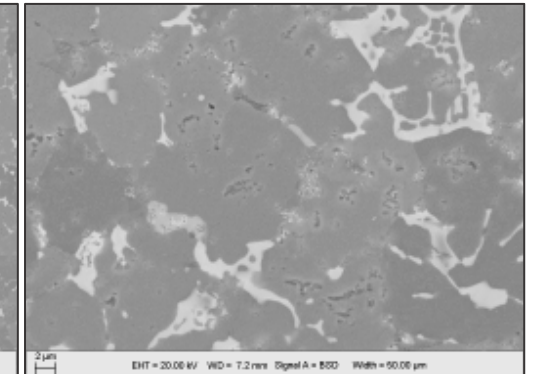
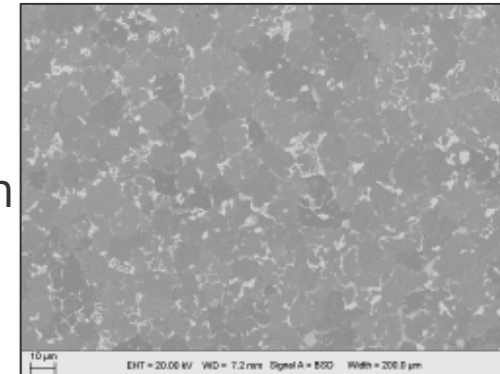
With increasing build height, microstructure coarsens (decreasing cooling rate) and amount of Ti-rich constituent decreases



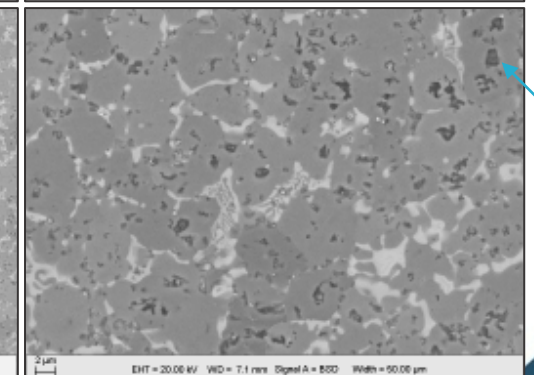
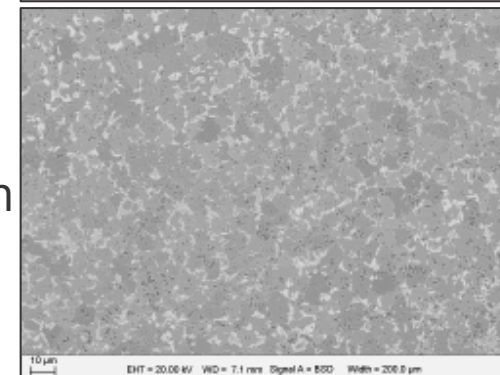
5.5 mm



4.5 mm



2.5 mm

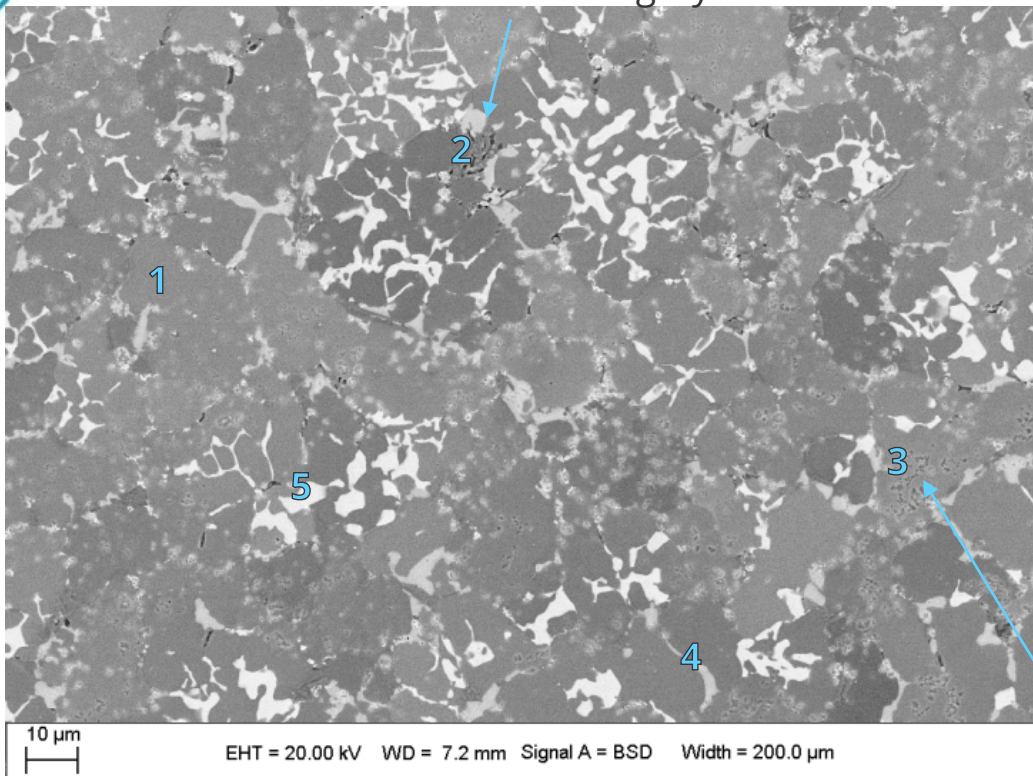


Ti-rich constituent (darkest)



Middle region: 2.5-5.5 mm build height

This dark gray constituent is attached to “medium gray”



1: Matrix = Si & Fe

2: **Darkest gray = Fe & ? + B**

3: Other dark gray, Ti & Nb + **B (B in highest concentration)**

4: Medium gray = Nb, Fe & Si ✓

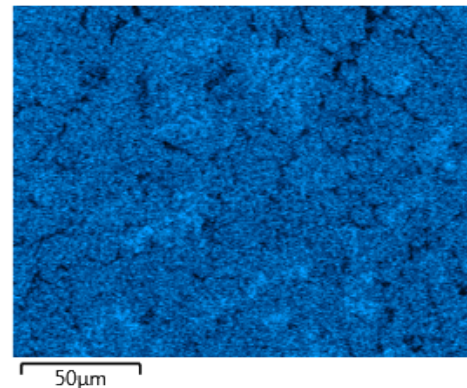
5: Bright = More Nb & Si, Less Fe ✓

This dark gray constituent (Ti-rich) is within the matrix

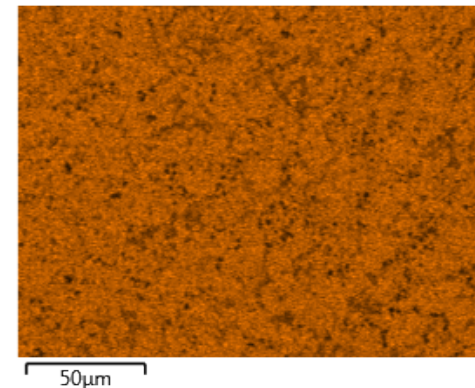
1st appearance of another dark constituent (labelled #2)

- Always attached to “medium gray”
- As shown in coming slides, this new dark constituent contains Fe, but is depleted in all the rest of the mapped elements
- Since the BSE contrast is dark, suggests light elements
- Perhaps B-rich?

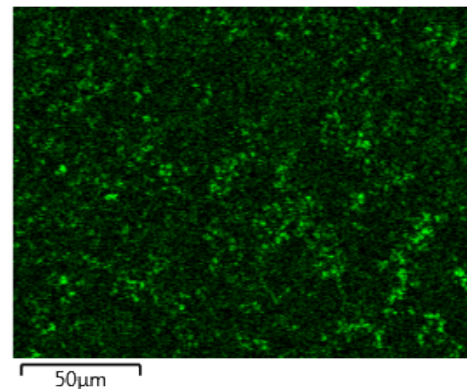
Si Kα1



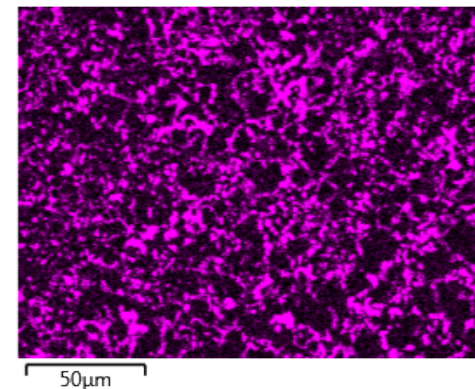
Fe Kα1



Ti Kα1

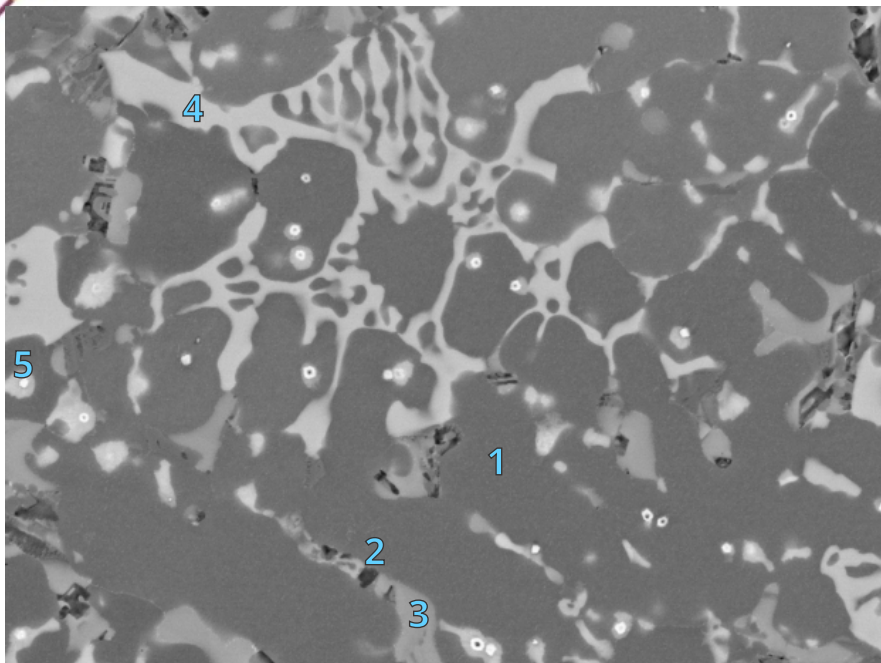


Nb Lα1





Top region – 9.5 mm



1: Matrix = Si & Fe

2: Darkest gray = Fe & ? **+ B**

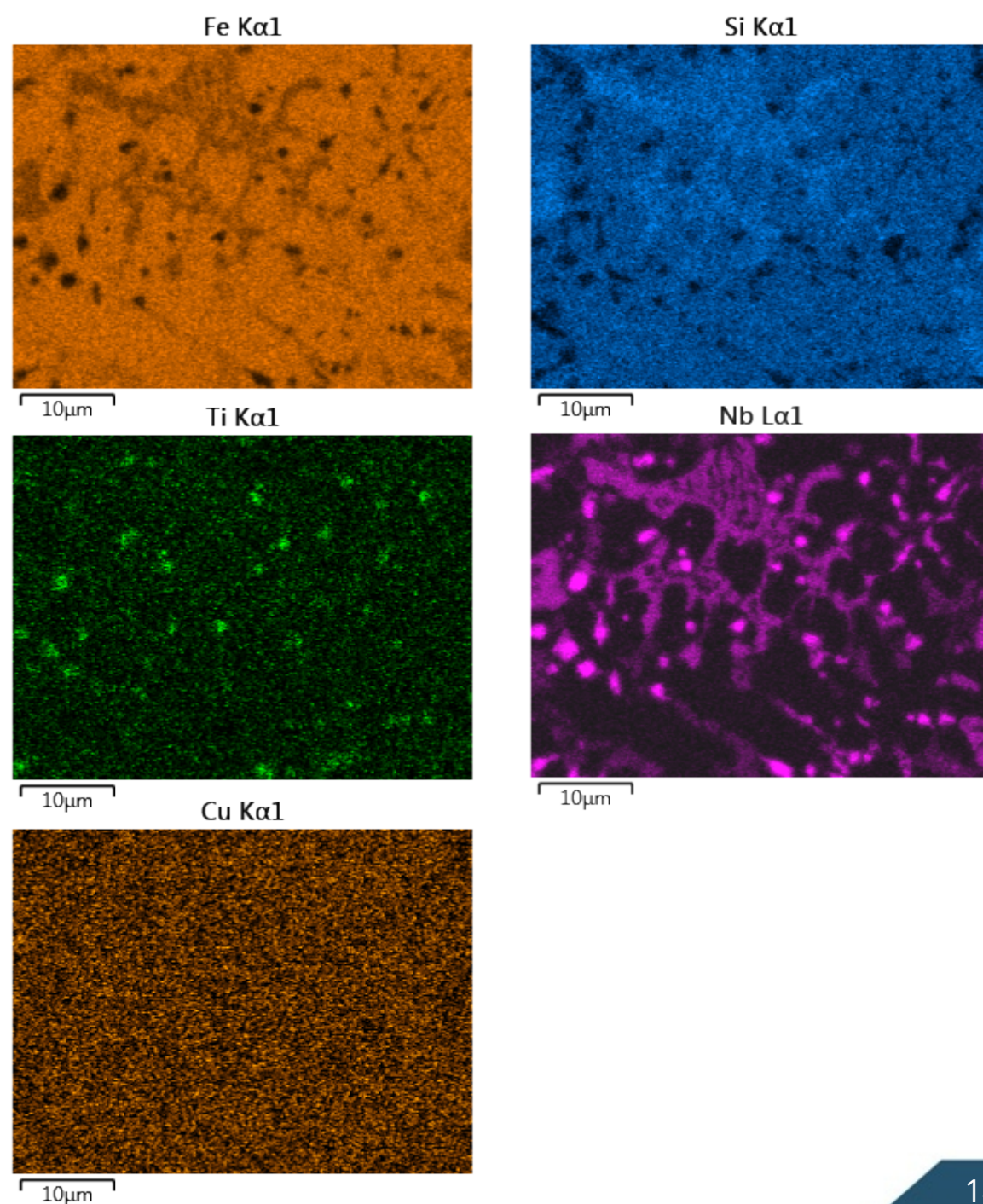
3. Medium gray = Nb, Fe & Si **+ B**

4: Light gray = More Nb & Si, Less Fe ✓

5: Bright spots = Nb & Ti **+ B** (this is where the most B is in this region)

***4 & 5 are sometimes indistinguishable in BSE**

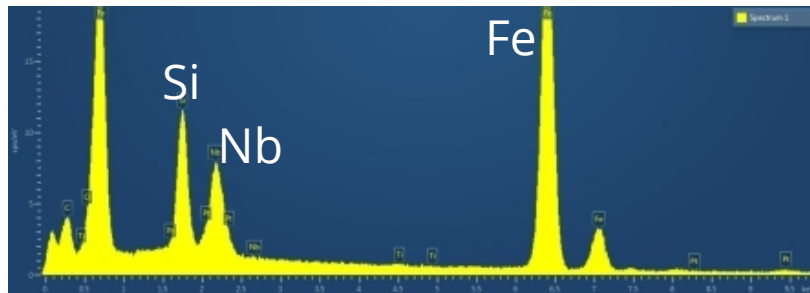
Main difference here is lack of Fe & Si in these regions



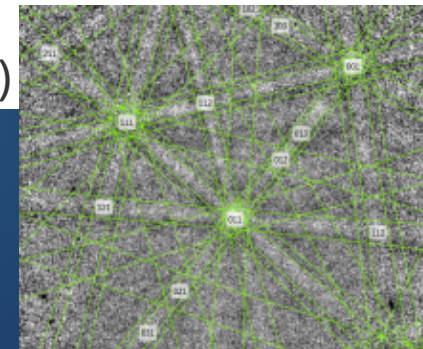
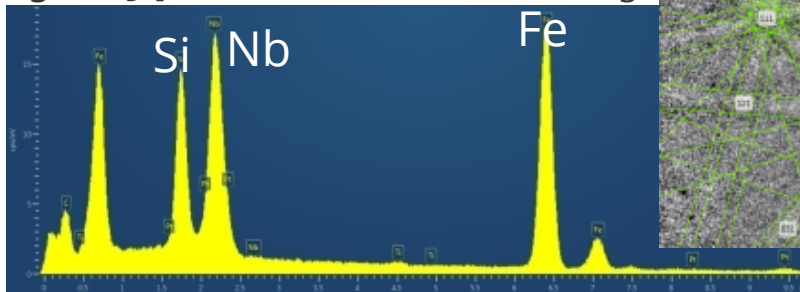


Summary of observed phases

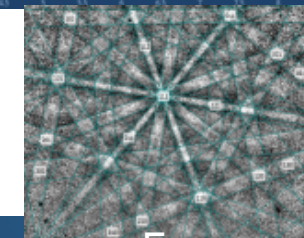
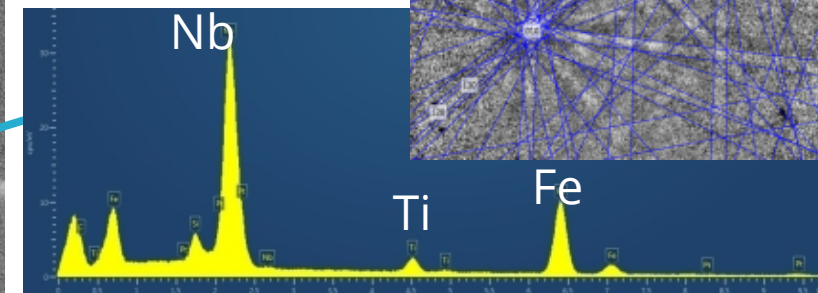
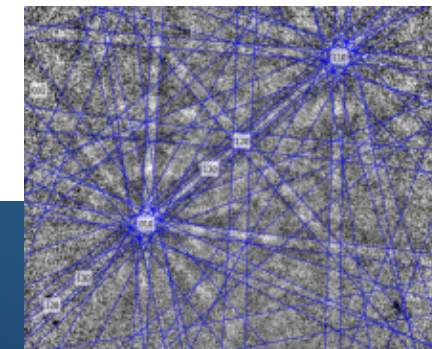
Laves Phase (Fe_2Nb)



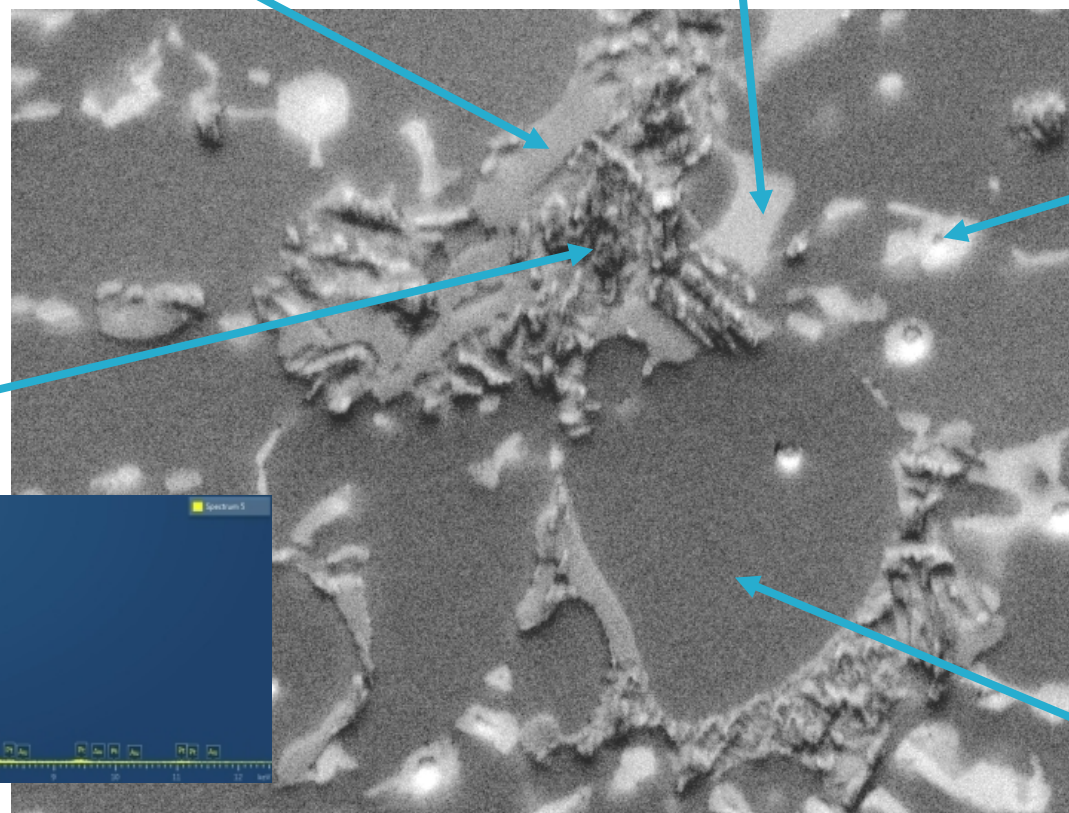
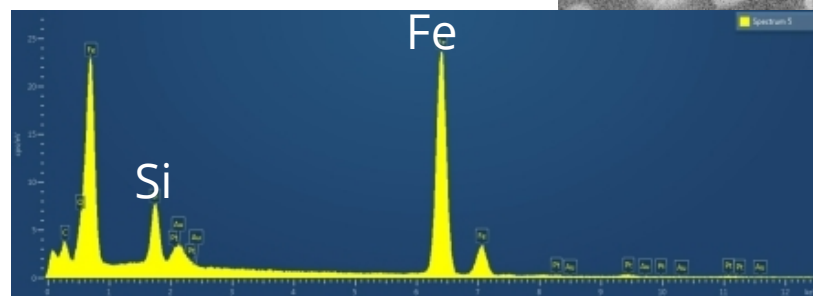
Fe_3Si -type (ordered BCC, DO_3)



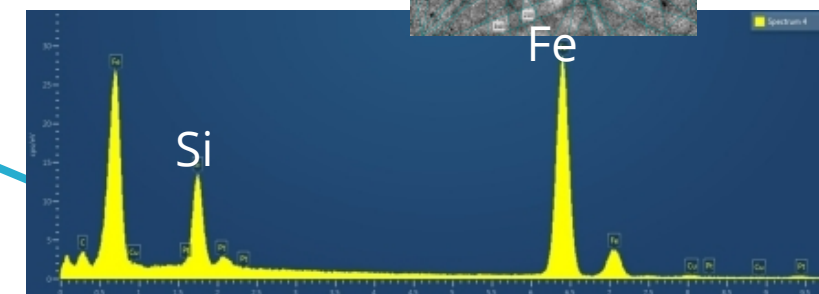
Ti/Nb-boride:
 TiNbB_4 or NbB_2
or TiB_2



Unknown

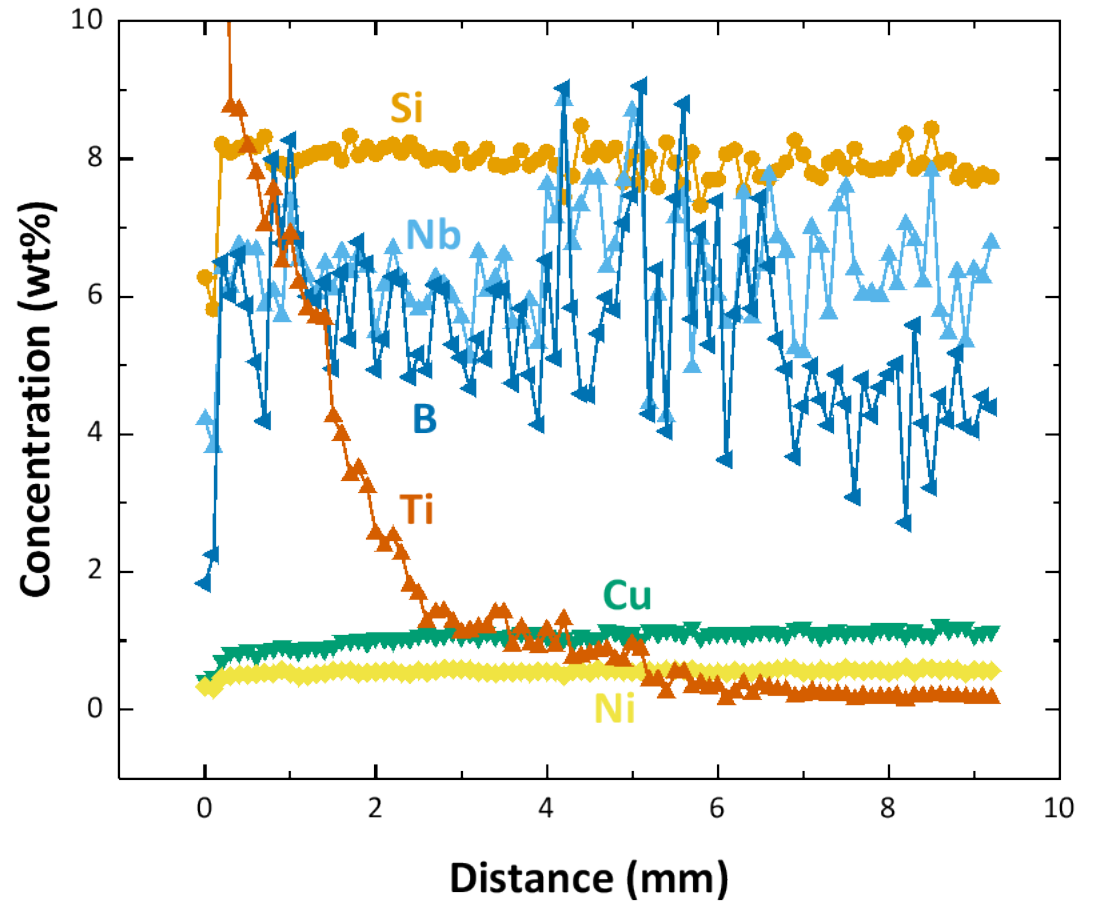
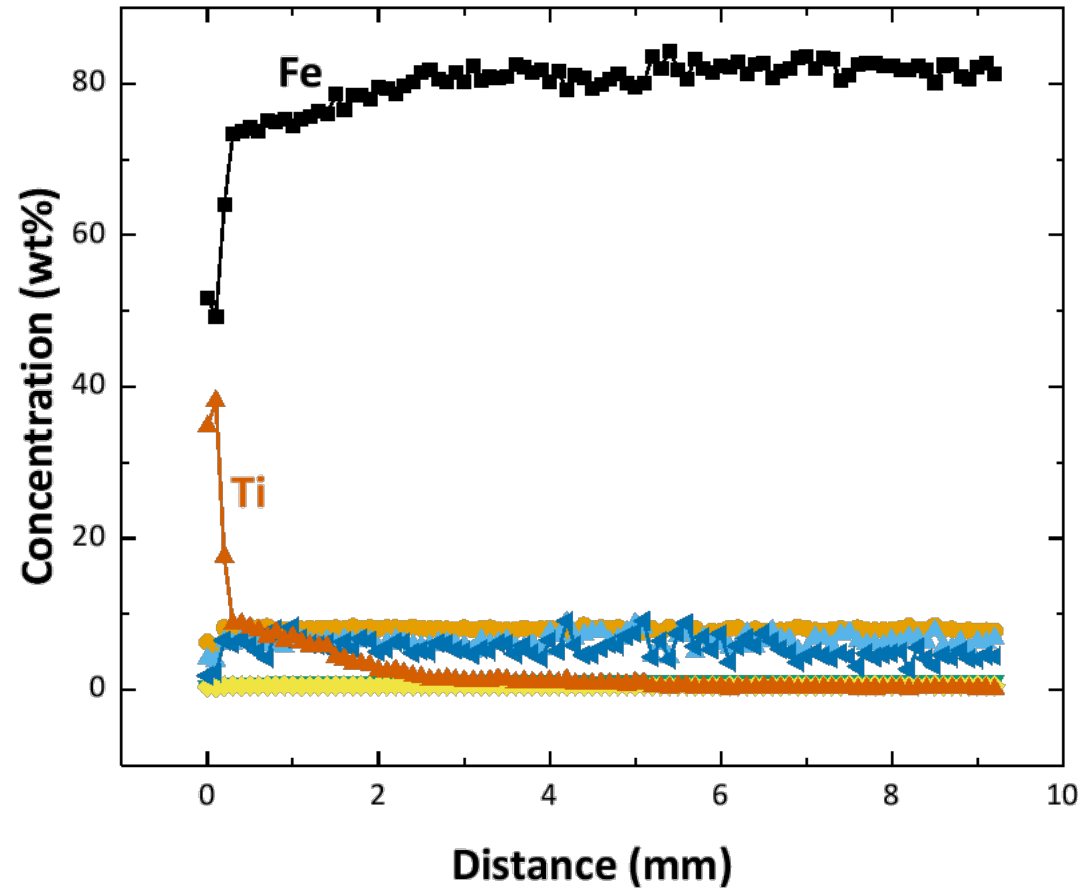


Matrix: BCC





Wavelength dispersive spectroscopy of entire build

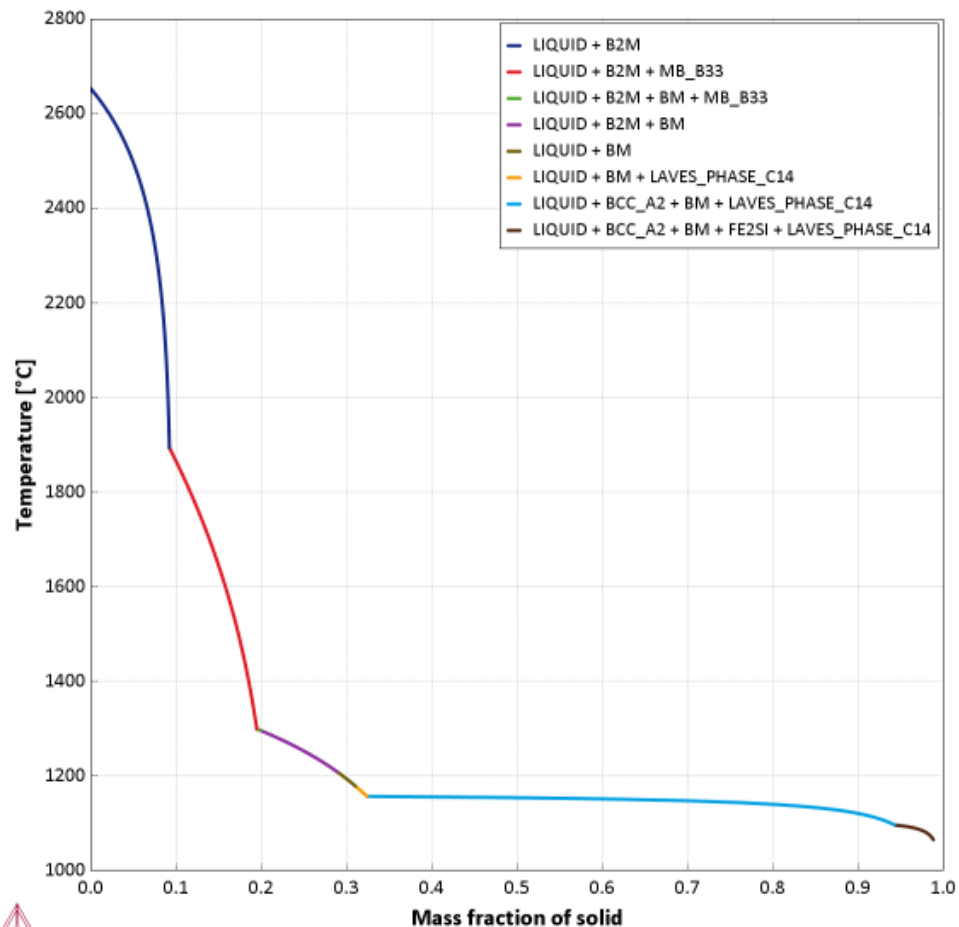




Thermo-calc Scheil Solidification Analysis

Bottom (Ti-rich) composition (In wt %):

70% Fe, 7.4% Si, **6.5% Ti**, 6.9% Nb, 0.8% Cu, 0.5% Ni, 7.8% B



B2M = Ti_2B

MB_B33 = $(\text{Fe,Ti,Nb,Ni})_1(\text{B})_1$ – so a MB boride

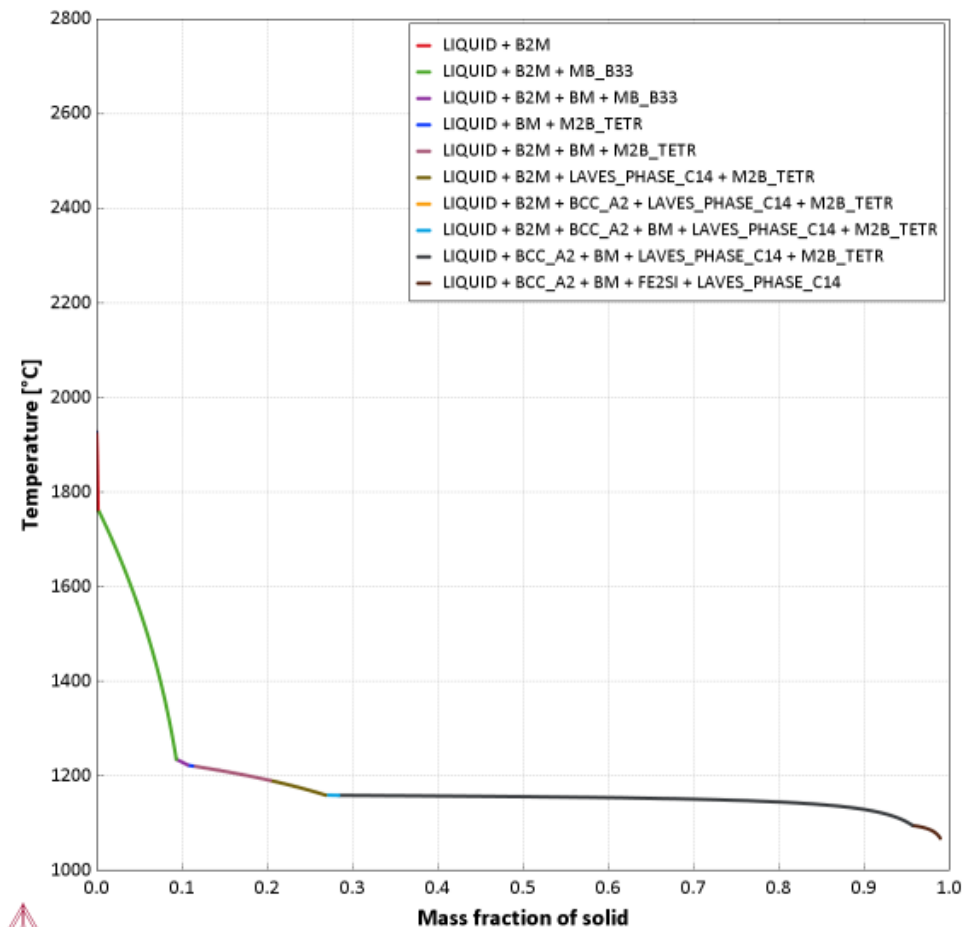
BM = $(\text{Fe,Ti})_1\text{B}_1$ – a different composition MB boride

LAVES_PHASE_C14 = $(\text{Fe,Si,Ti,Nb,Cu,Ni})_2(\text{Fe,Si,Ti,Nb,Cu,Ni})_1$ – the hexagonal C14 type Laves phase, Fe_2Nb

M₂B_TETR = $(\text{Fe,Ni})_{0.67}(\text{B})_{0.33}$

Top composition (In wt %):

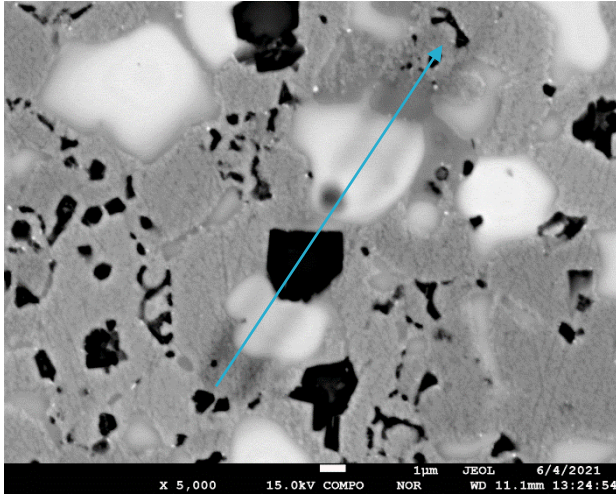
80% Fe, 7.7% Si, **0.2% Ti**, 6.2% Nb, 1.1% Cu, 0.2% Ni, 4.6% B



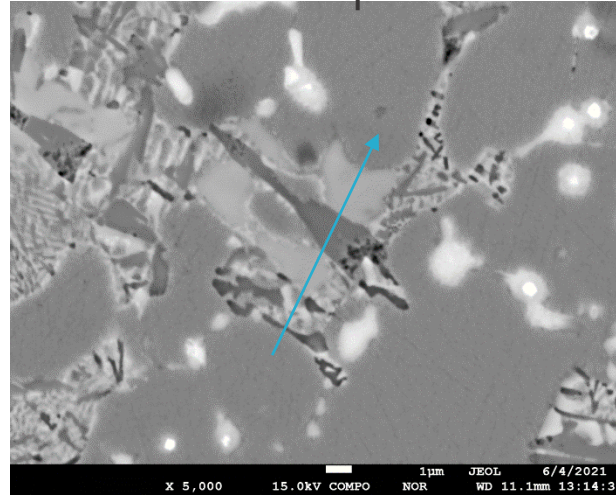


Lave phase throughout sample is similar

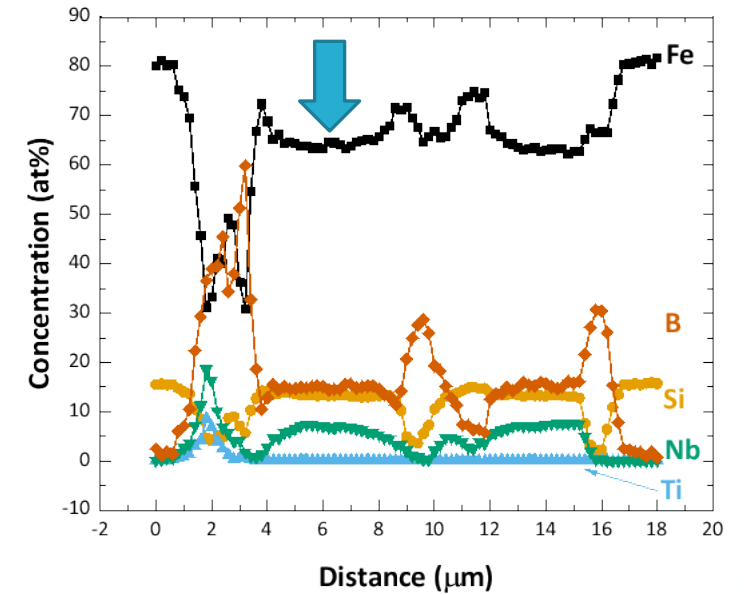
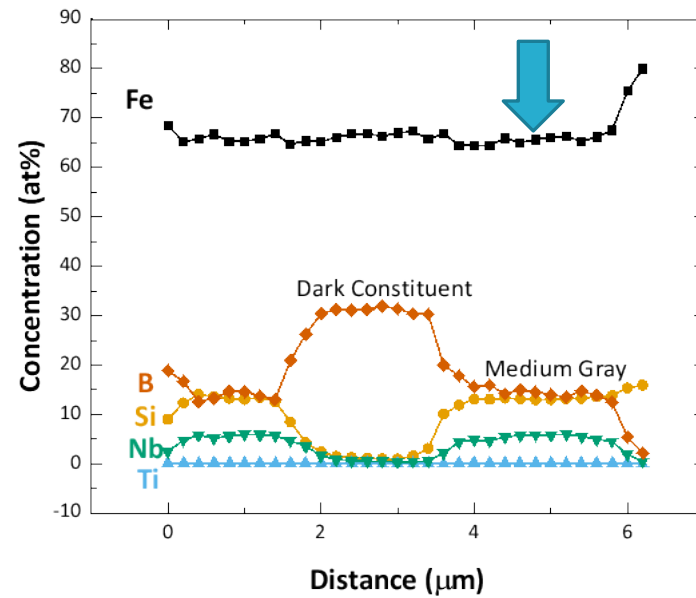
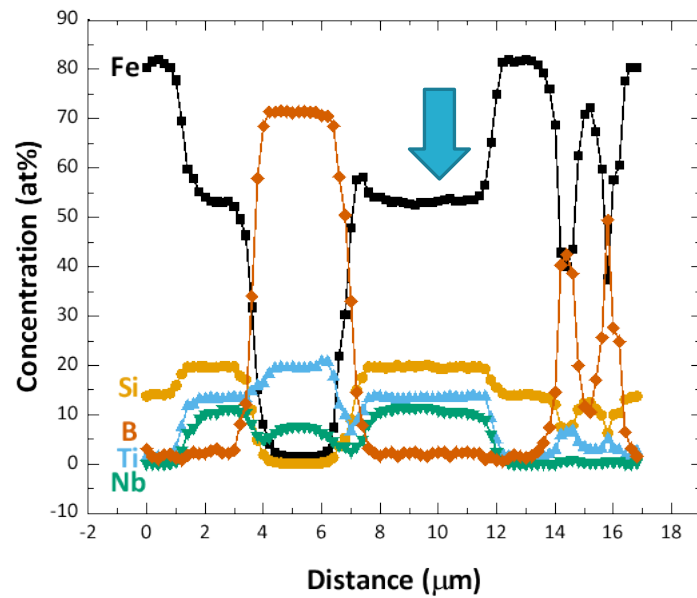
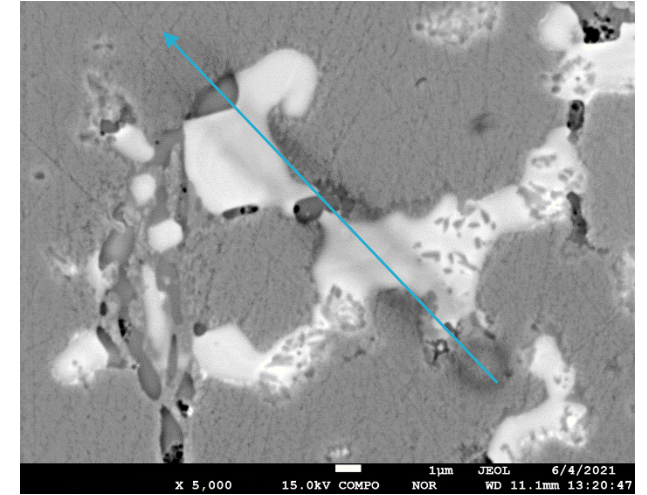
Bottom



Top



Middle

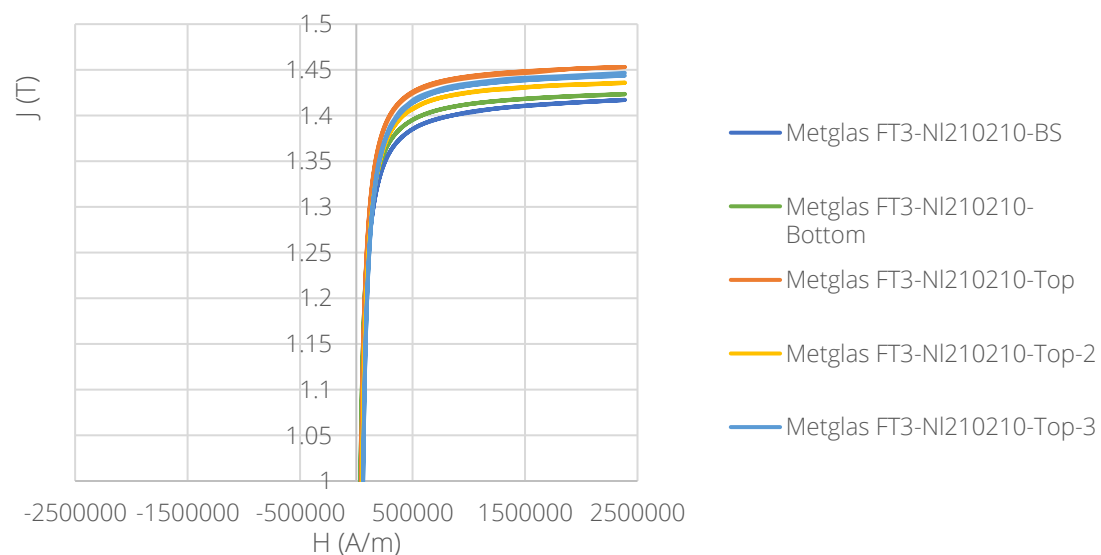




Magnetic Properties (VSM measurement)

Sample ID	Volume (m ³)	Density (g/cm ³)	mass (g)	Msat (AVG) (A/m)	Jsat (AVG) (T)	Coercitivity (AVG) (A/m)	Susceptibility Average	Permeability Average
Metglas FT3-NI210210-BS	2.00E-09	7.53E+00	1.50E-02	1.09E+06	1.37E+00	4.55E+02	37.57	38.57
Metglas FT3-NI210210-Bottom	2.00E-09	7.51E+00	1.50E-02	1.10E+06	1.38E+00	3.85E+02	36.47	37.47
Metglas FT3-NI210210-Top	2.83E-09	7.67E+00	2.17E-02	1.16E+06	1.45E+00	4.13E+02	25.88	26.88
Metglas FT3-NI210210-Top-2	3.56E-09	7.65E+00	2.72E-02	1.14E+06	1.44E+00	4.14E+02	19.46	20.46
Metglas FT3-NI210210-Top-3	4.14E-09	7.66E+00	3.17E-02	1.15E+06	1.44E+00	4.27E+02	16.79	17.79

Metglas FT3

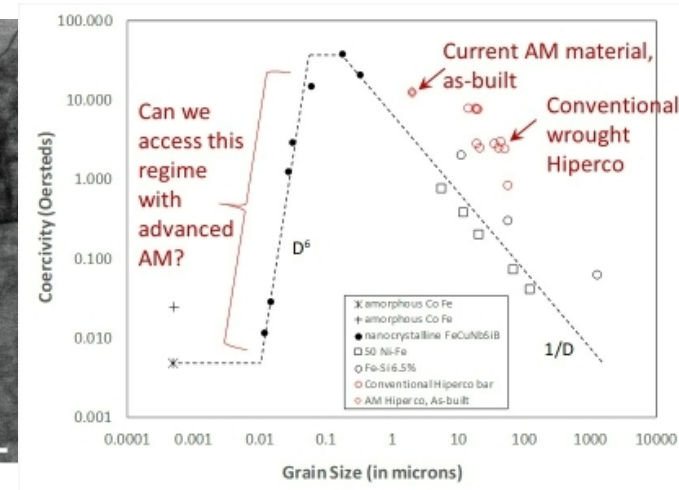
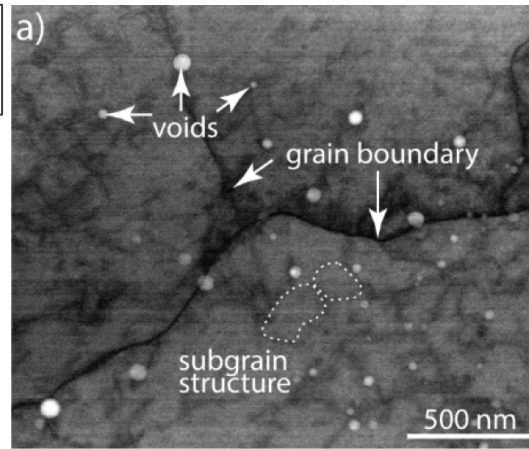
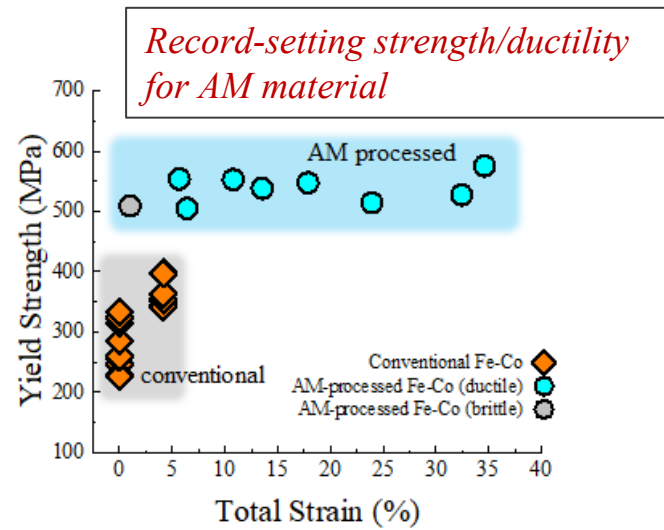




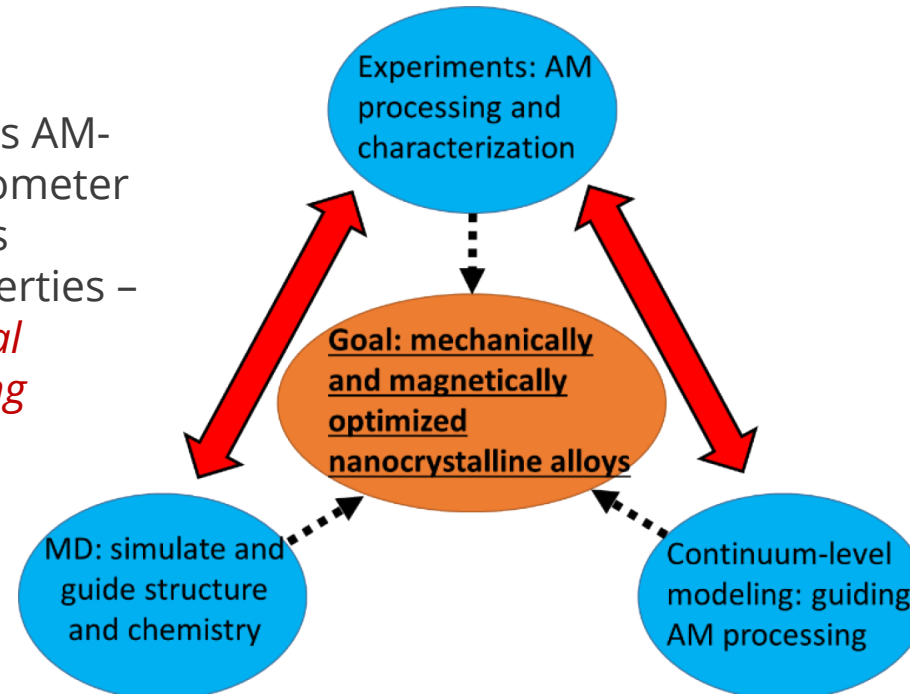
Summary

1. Printed a novel customized FeSiNbBCu soft magnetic alloys from milled powder-like flakes using LB-DED processing
2. Microstructure had coarse grain size with up to five distinct phases
3. Ti “contamination” was observed throughout build height due to gettering of the B-content; forming (Ti,Nb) boride precipitates – need to consider build plate material!
4. Resultant microstructure phase evolution was supported by Thermcalc predictions
5. Magnetic properties were slightly impacted by varying Ti-content, most notably the full-field induction and relative permeability values; the former decreasing and the latter increasing with increased Ti.

Project purpose and objectives

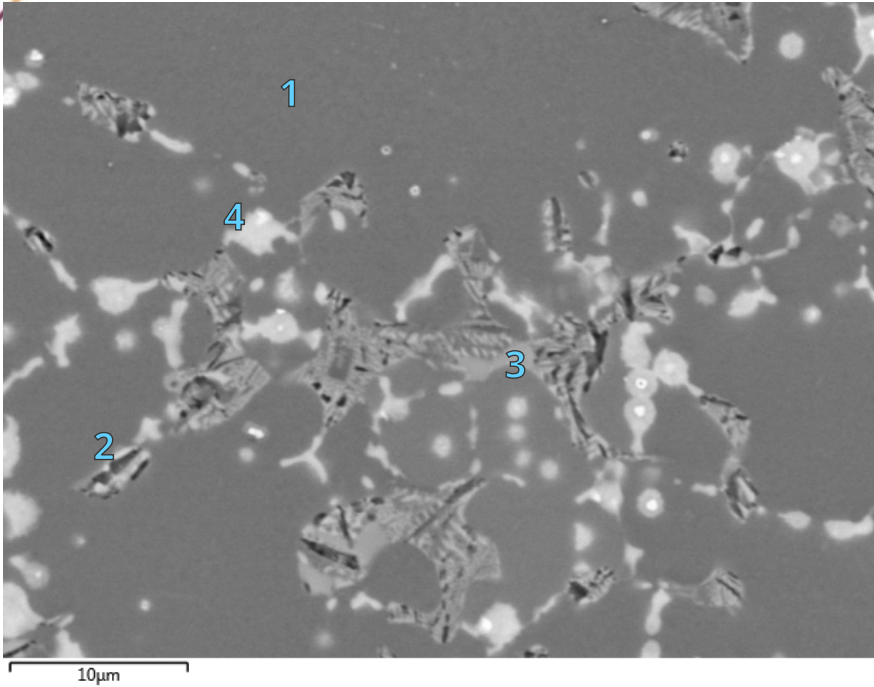


Previous research suggests AM-enabled hierarchical (nanometer-to-micrometer) structures enhance mechanical properties – *route to improve mechanical properties without sacrificing magnetic performance?*





Top region – 7.5mm and higher



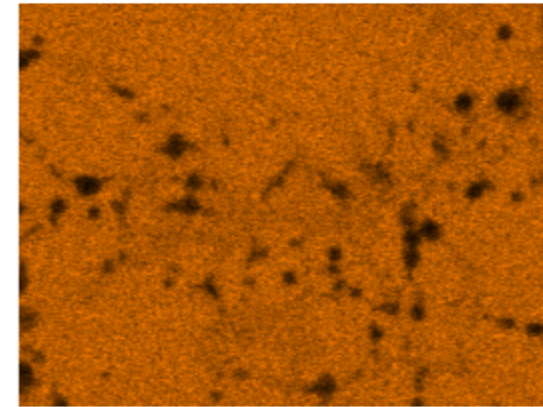
1: Matrix = Si & Fe

2: Dark = Fe & ?

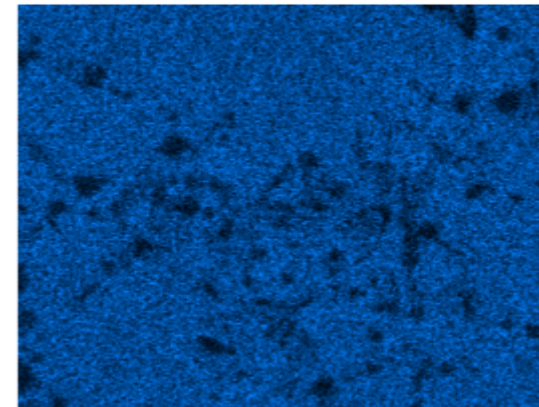
3: Medium Gray = Fe, Si, & Nb

4: Bright = Nb & Ti

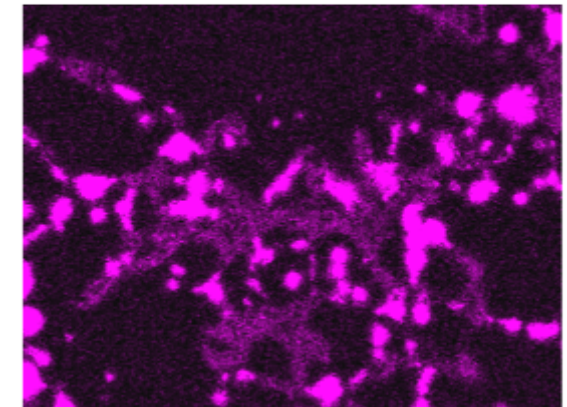
Fe K α 1



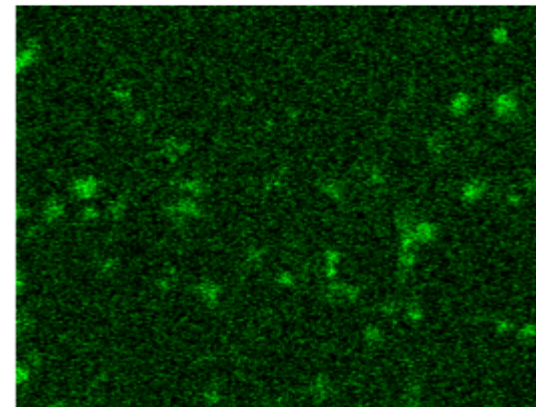
Si K α 1



Nb L α 1



Ti K α 1



Cu K α 1

