Precipitation strengthened CCAs: Combinatorial Screening & Trends in the AlCrFeNiTi System

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- Motivation
- Combinatorial Screening Results → RWTH AACHEN UNIVERSITY | MATERIALS CHEMISTRY
  - AlCoCrFeMn System
  - AlCrFeNiTi System
- Preliminary Studies in the AlCrFeNiTi System
- Alloy List
- Arc Melted Characterization Results
  - XRD
  - SEM Microstructures and EDS Analysis
  - DSC and High Temp XRD
  - TEM (Sample B15)
- Arc Melted Sample Summary
- Vacuum Induction Cast Results
  - XRD
  - SEM Microstructures and EDS Analysis
  - Compression Test Results
- Future Work

Motivation – High Temperature Structural Materials

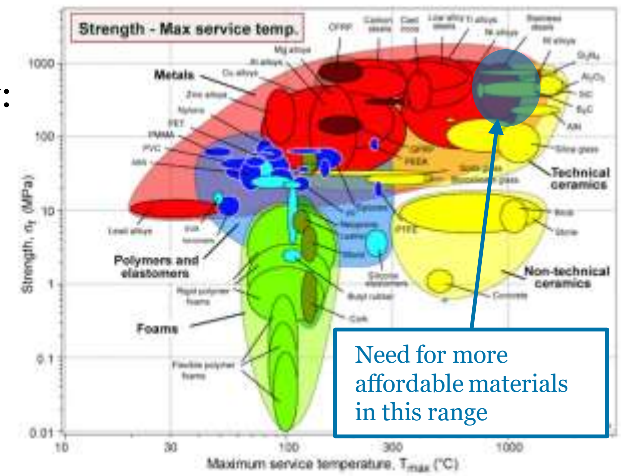
Steam turbines are used to generate ~80% world's energy

http://www.mechscience.com/steam-turbine-introduction-and-principle-operations/

Ideal alloys would have:

- Low-cost & low-density: **<7 g/cm³**
- Creep resistance up to **900°C**
- Maximized solid solution strengthening & precipitation hardening

Novel high temperature materials needed!

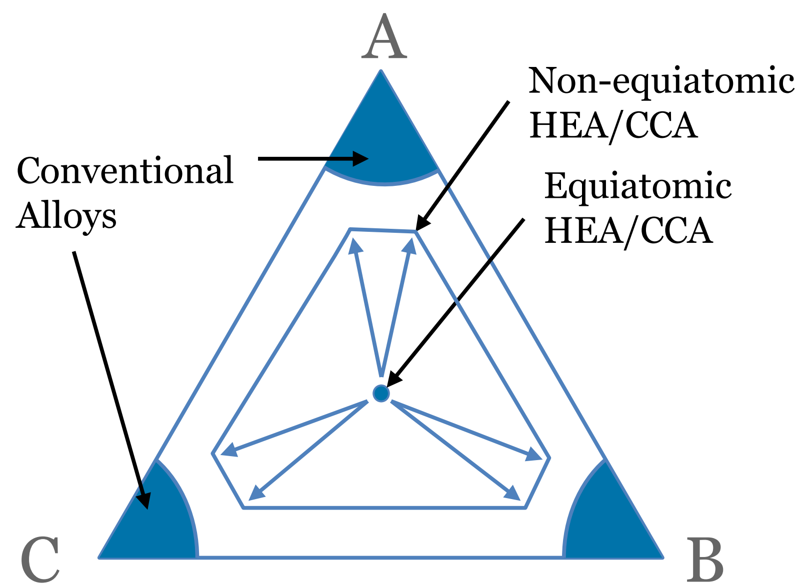


https://commons.wikimedia.org/wiki/File:ASHBY_D.png

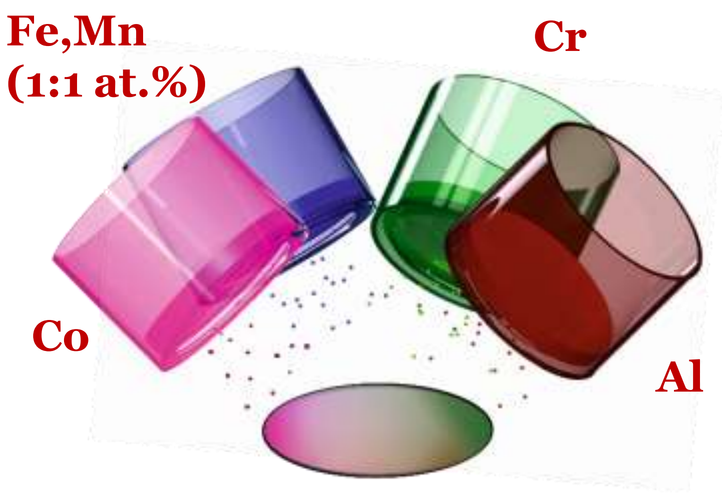
Where and how do we search for new materials that fill the gaps between conventional alloy classes??

"High entropy alloys (HEAs) and … [compositionally complex] alloys (CCAs) open alloy development to a vast richness of complexity." [1]

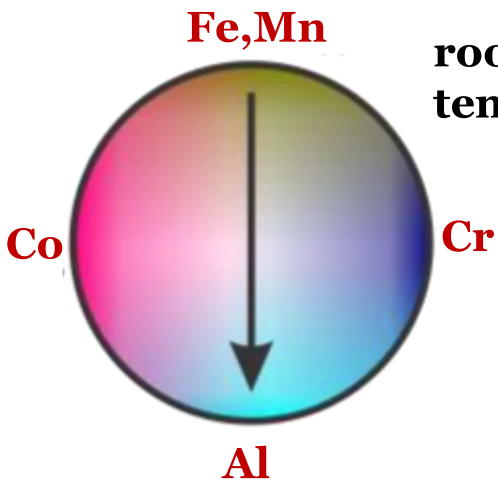
Miracle, Daniel, et al. "New strategies and tests to accelerate discovery and development of multi-principal element structural alloys." Scripta Materialia 127 (2017): 195-200.



Combinatorial Synthesis

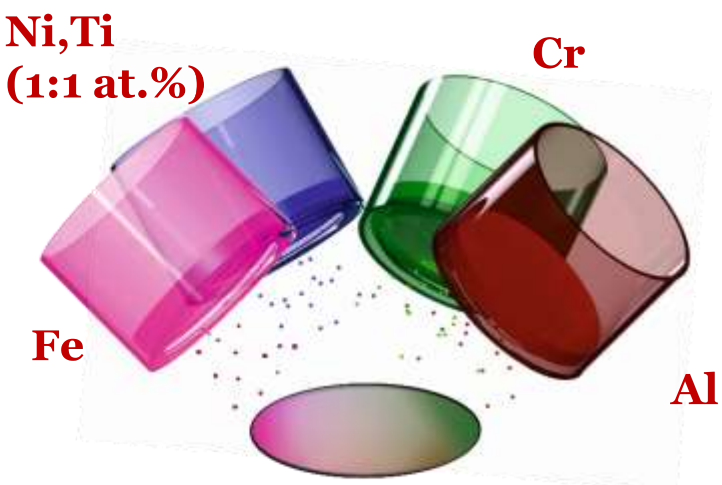


AlCoCrFeMn

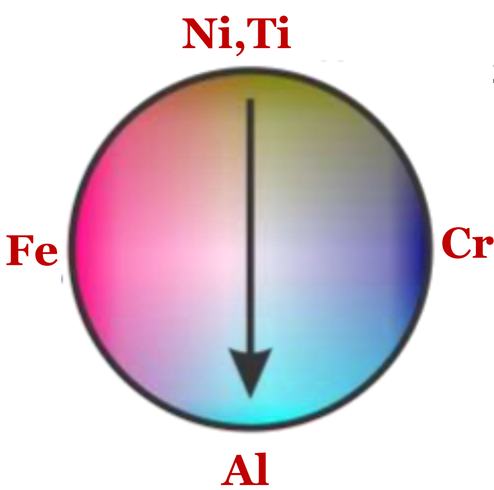


room temperature

$Al_{3\text{-}55\ at.\%}Co_{20}Cr_{20}(FeMn)_{60\text{-}Al}$



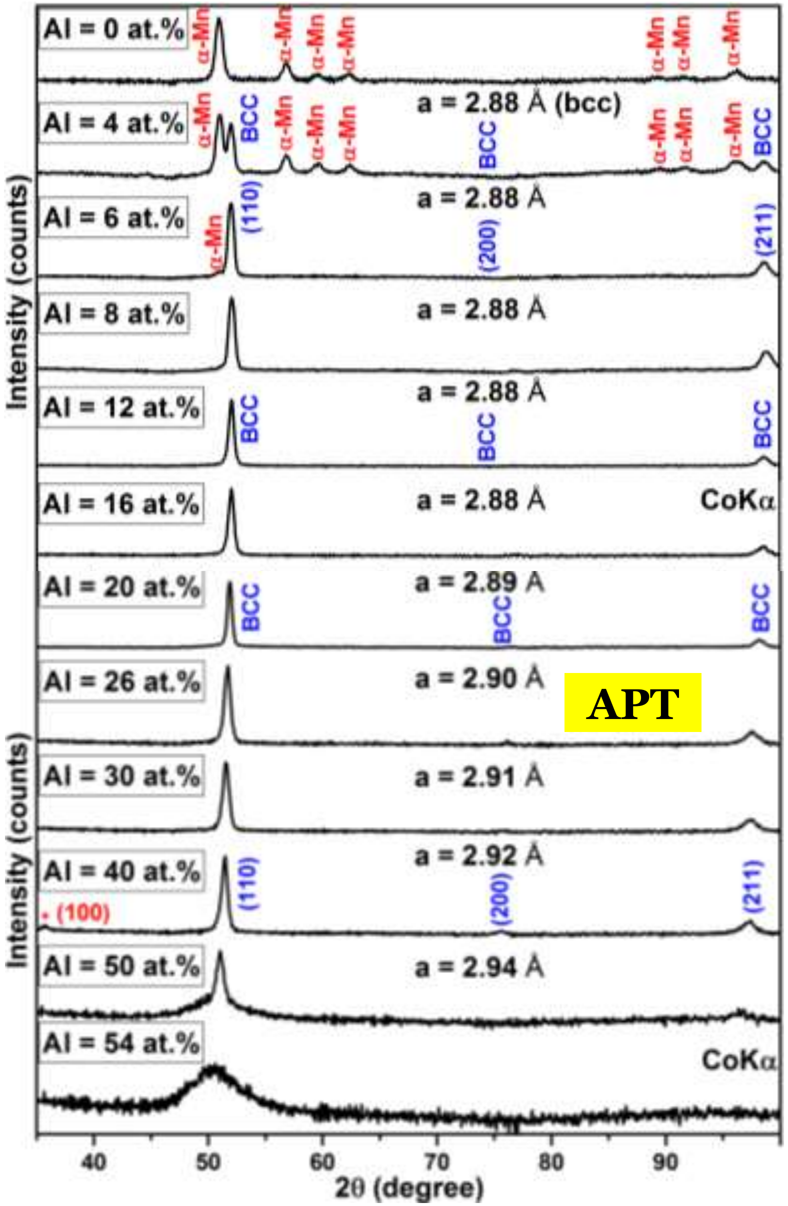
AlCrFeNiTi

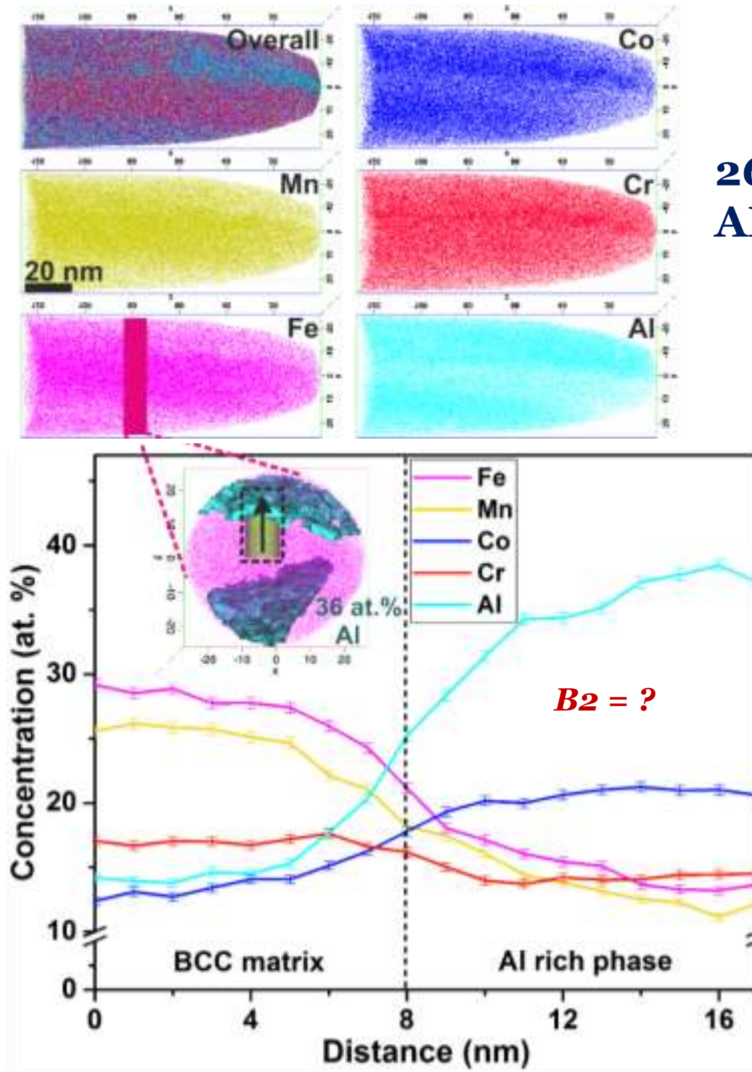


200 °C

$Al_{6\text{-}35\ at.\%}Cr_{20}Fe_{35}(NiTi)_{45\text{-}Al}$

AlCoCrFeMn thin film - Phase Formation

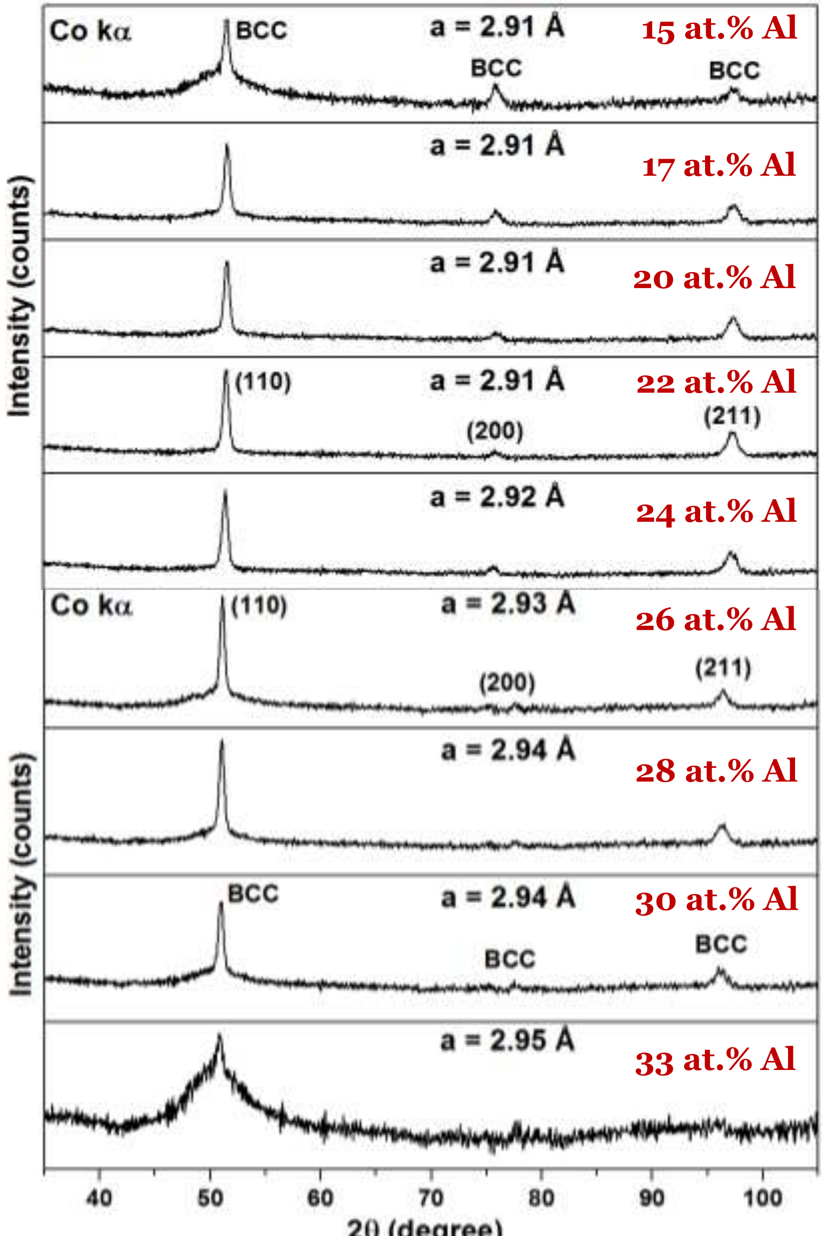




26 at.% Al APT

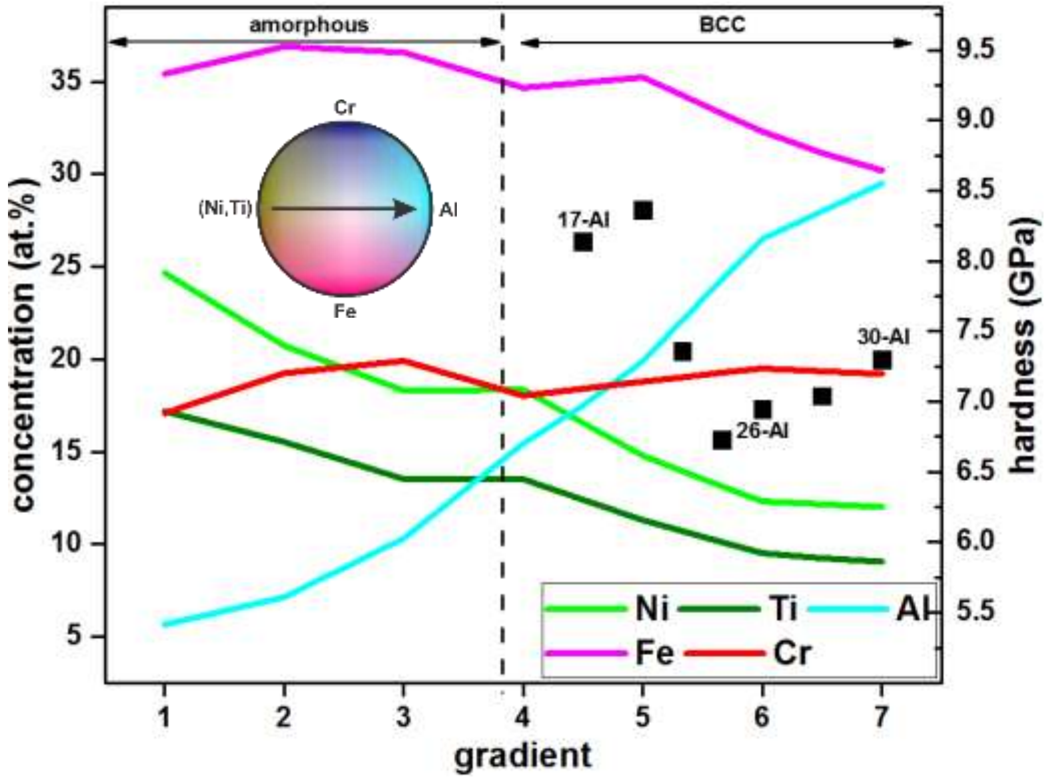
- AlCo based nano-separation seen at 26 at.% Al concentration

AlCrFeNiTi – Phase Formation



< 13 at.% Al containing thin film → amorphous
(limited kinetics during synthesis)

→ high temperature thin film synthesis in future



Bulk Studies of AlCrFeNiTi

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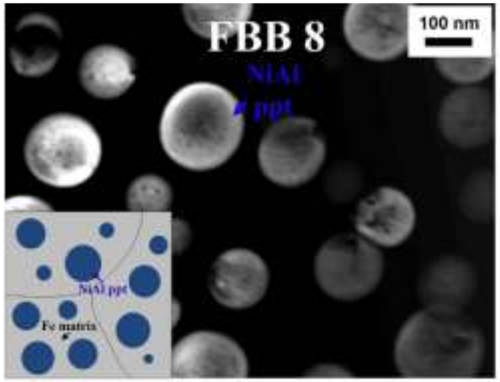
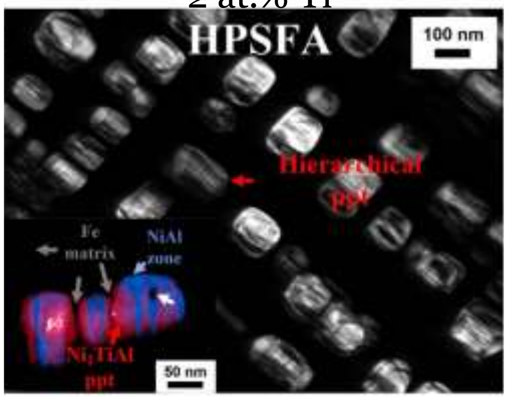
Preliminary Studies in the AlCrFeNiTi System

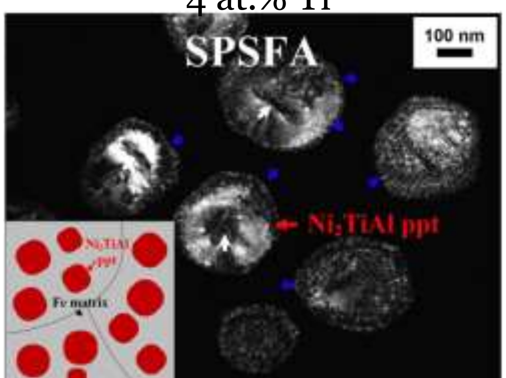
0 at.% Ti



2 at.% Ti



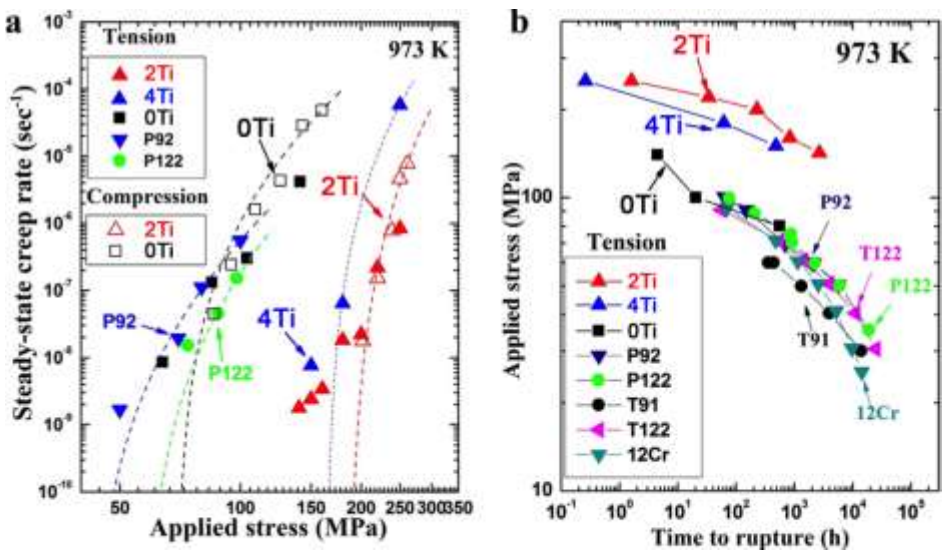
4 at.% Ti



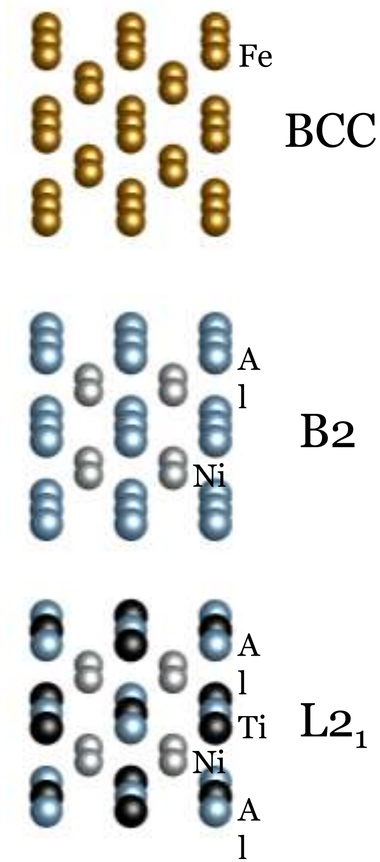
Inspiration: Fe-base Superalloys

FBB8 Alloy:

- Fe-10Cr-10Ni-6.5Al-3.5Mo (minor Zr and B additions)
- Coherent B2 precipitates in disordered BCC matrix – Ferritic analogue to Ni-base superalloys
- Addition of Ti stabilizes $L2_1$ phase, greatly improving creep resistance

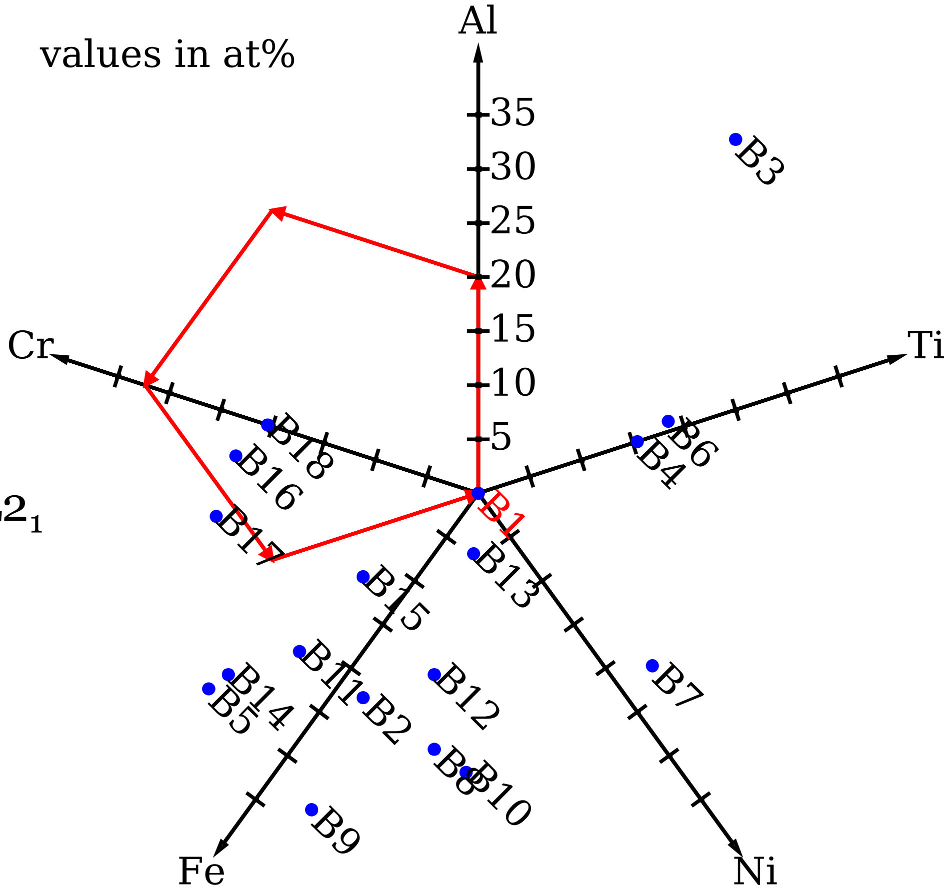


Song, Gian, et al. "Ferritic alloys with extreme creep resistance via coherent hierarchical precipitates." *Scientific reports* 5 (2015).



How far can creep resistance be improved by increasing $B2/L2_1$ phase fractions?

Goal: BCC matrix with B2 and/or $L2_1$ precipitates

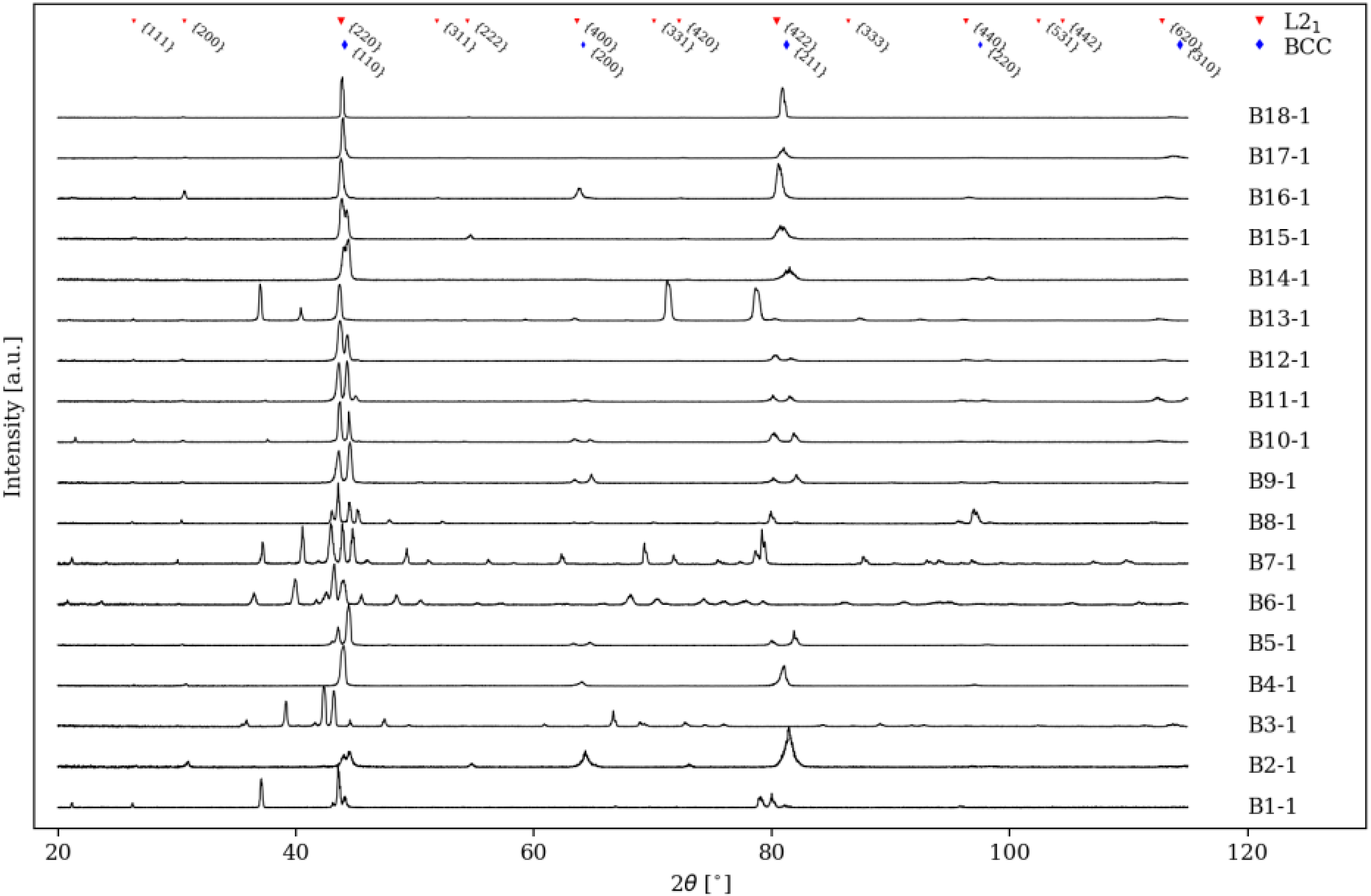


Design Guidelines:

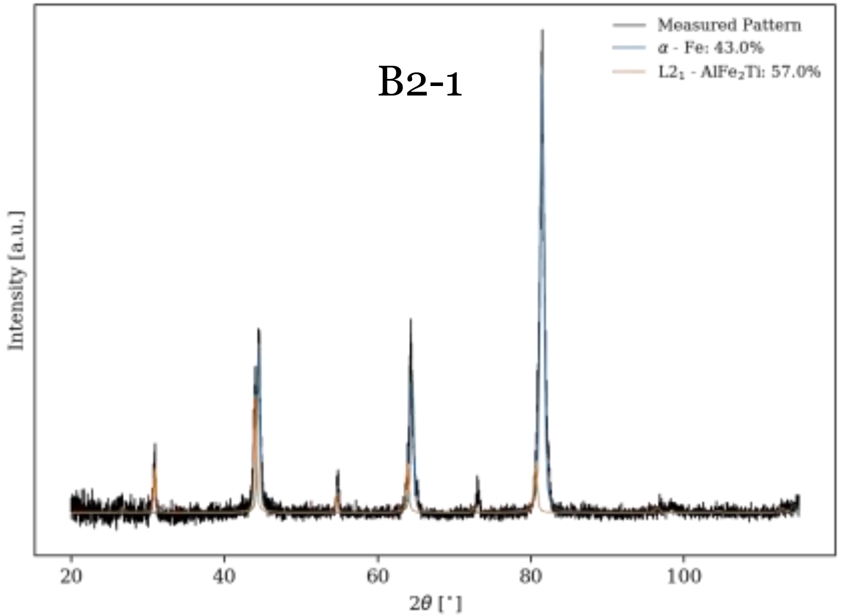
- Cheap: maximize Fe
- Lightweight: maximize Al, Ti
- Creep Resistant: include Ni, Ti for $L2_1$
- Oxidation Resistant: include Al, Cr
- Want large sampling area

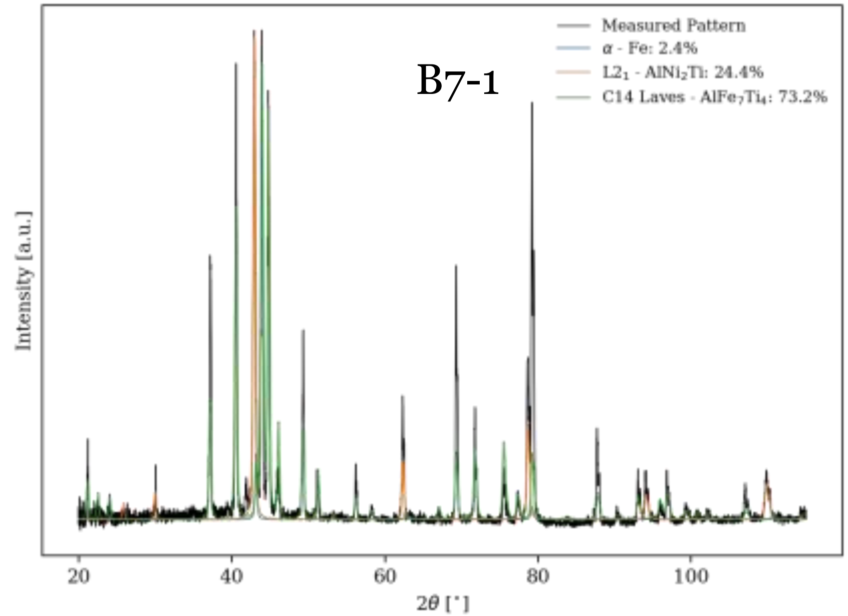
- So far 18 samples have been arc-melted and characterized
- Results will be compared to thin film libraries for high throughput validation

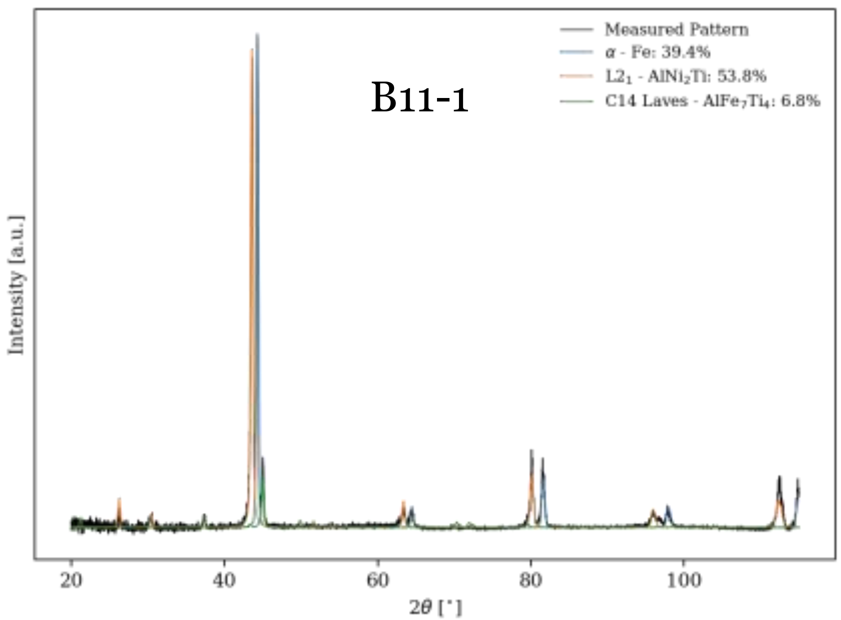
XRD Results – Arc Melted Samples

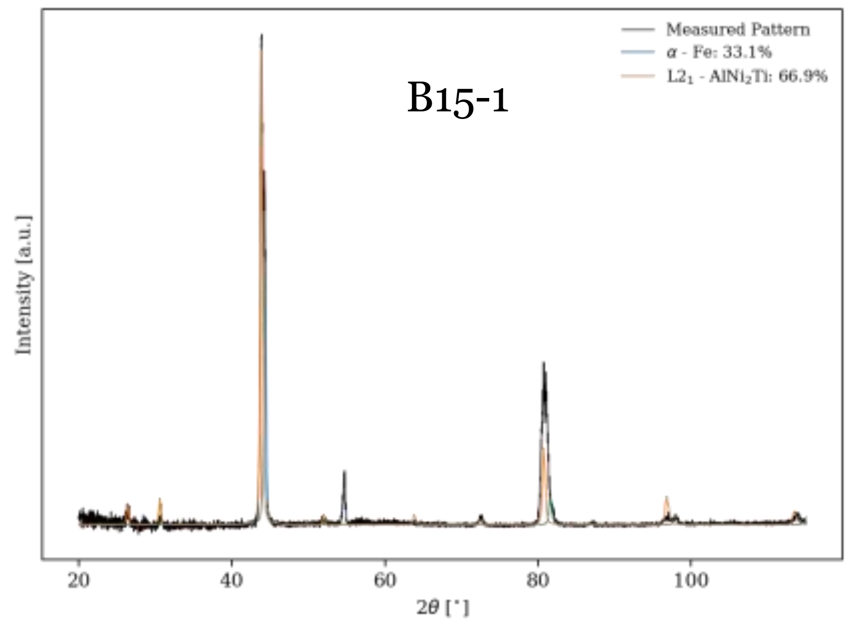


XRD Results – Rietveld Refinement

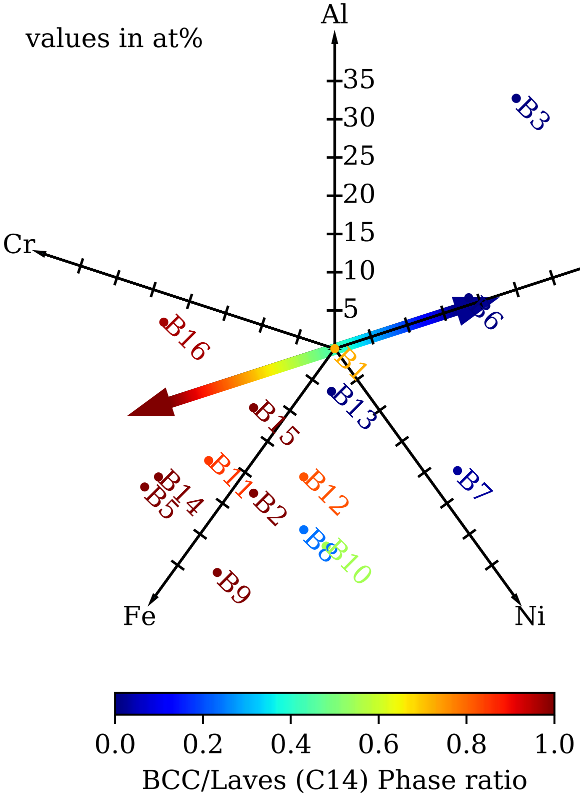








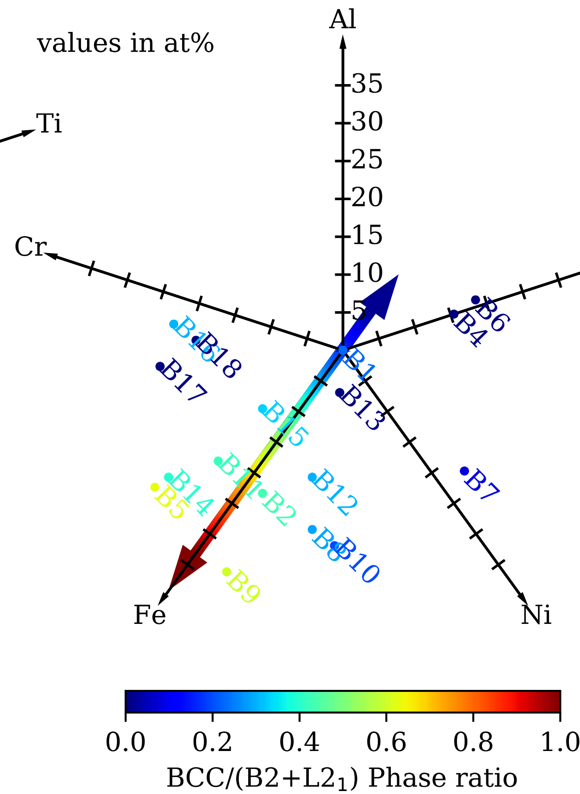
BCC-Laves (C14) Formation



BCC ratio increases along Fe/Cr axis

Laves ratio increases along Ti axis

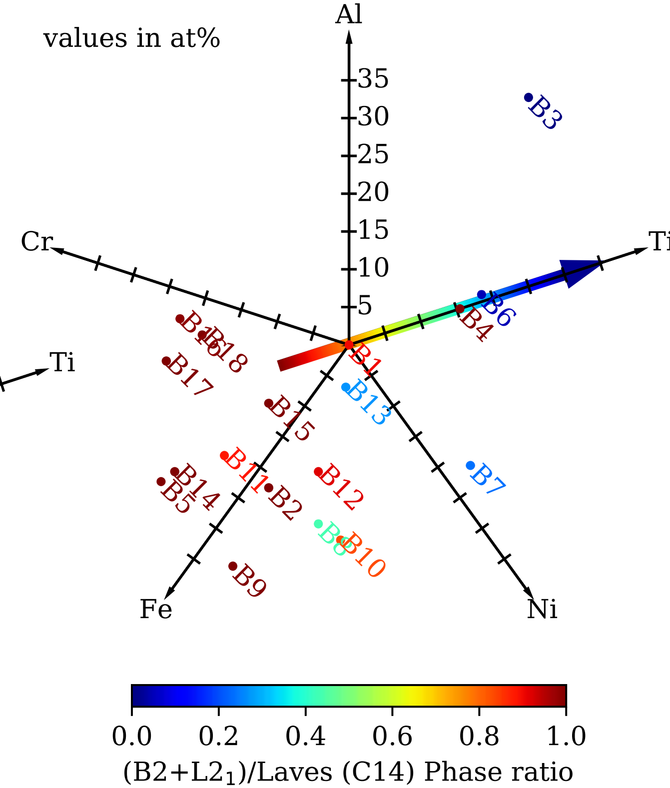
BCC-(B2+L2$_1$) Formation



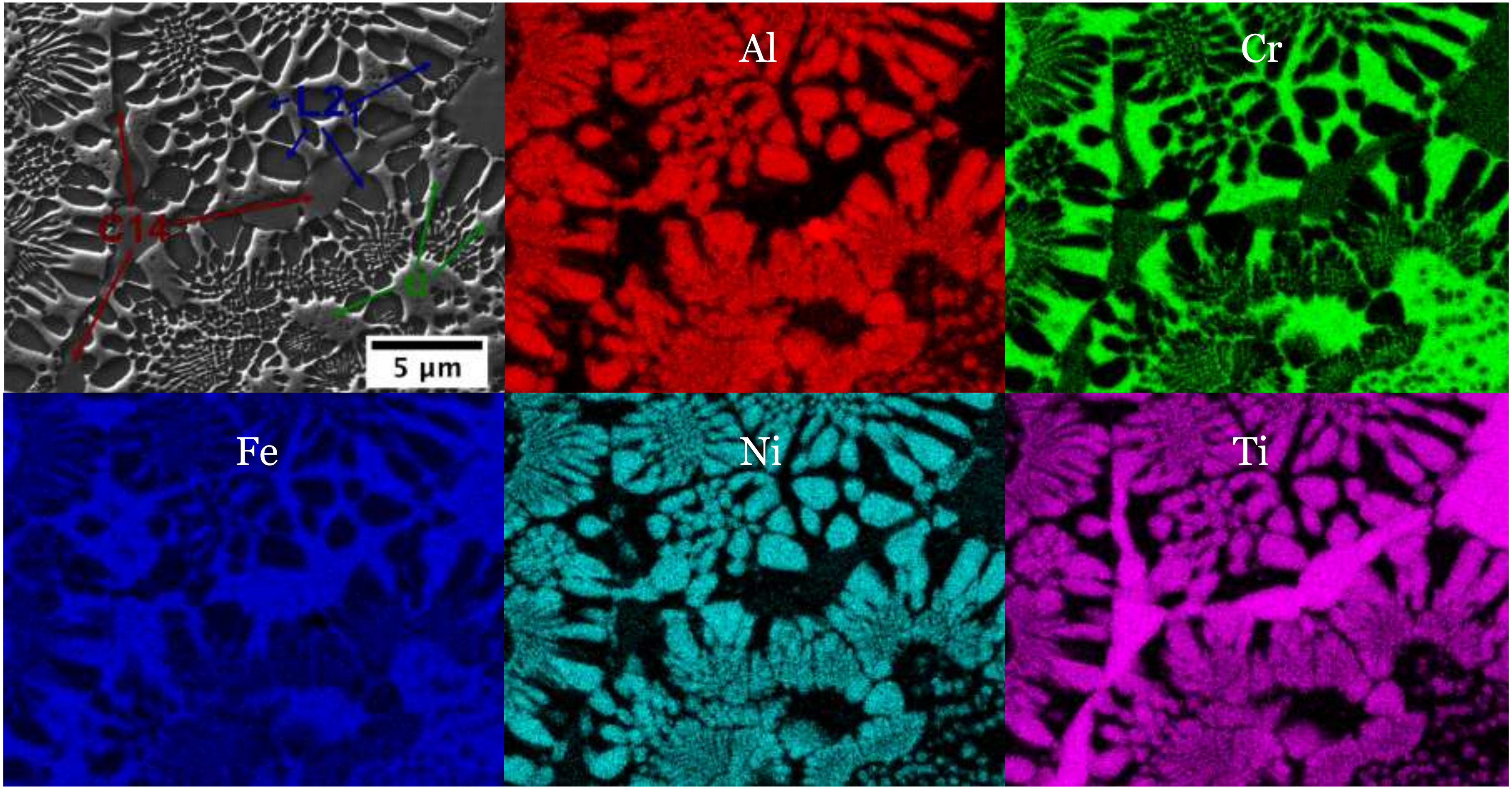
BCC ratio increases along Fe axis

(B2+L2$_1$) ratio increases along Al/Ti axis

(B2+L2$_1$)-Laves (C14) Formation

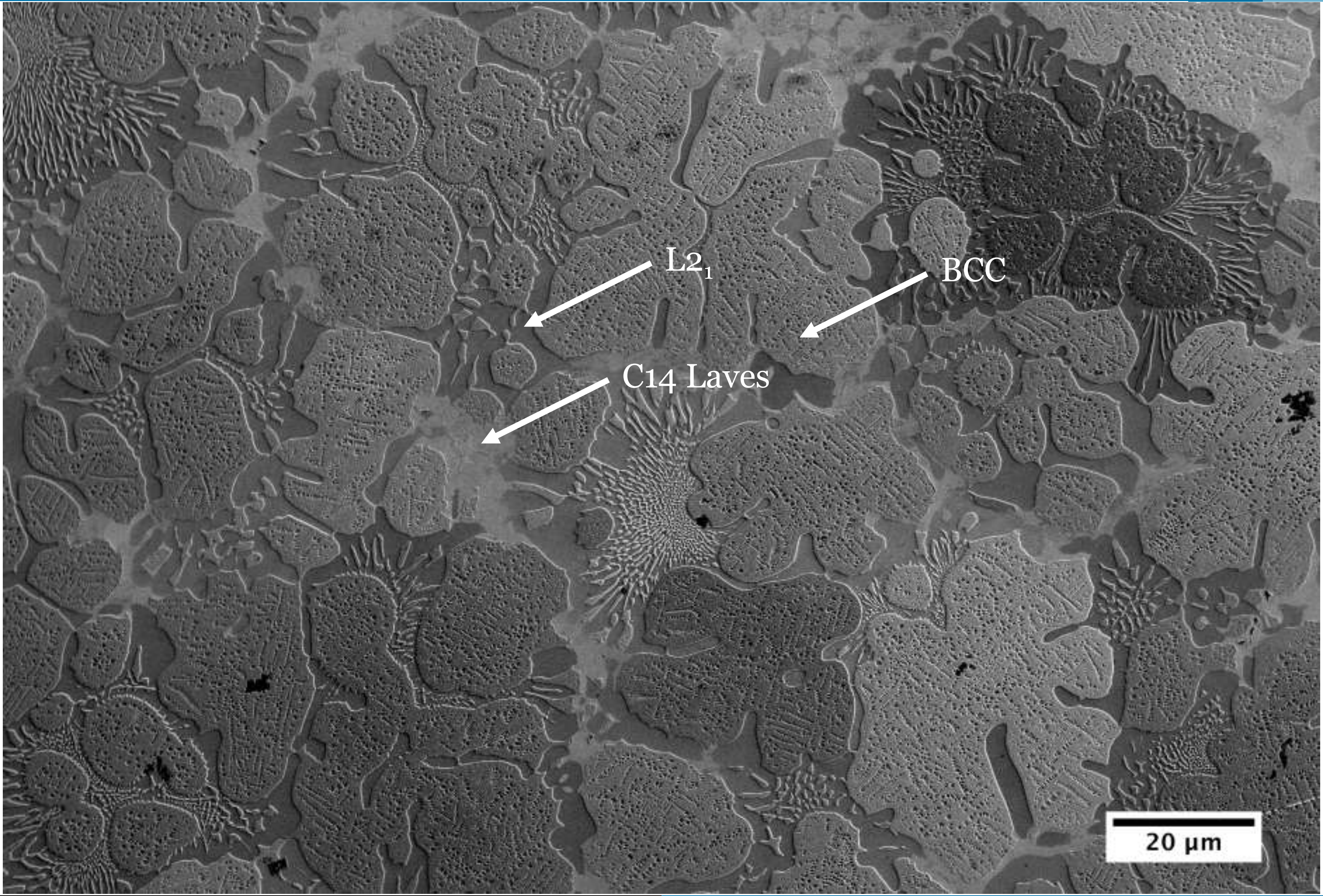


Laves ratio increases along Ti axis

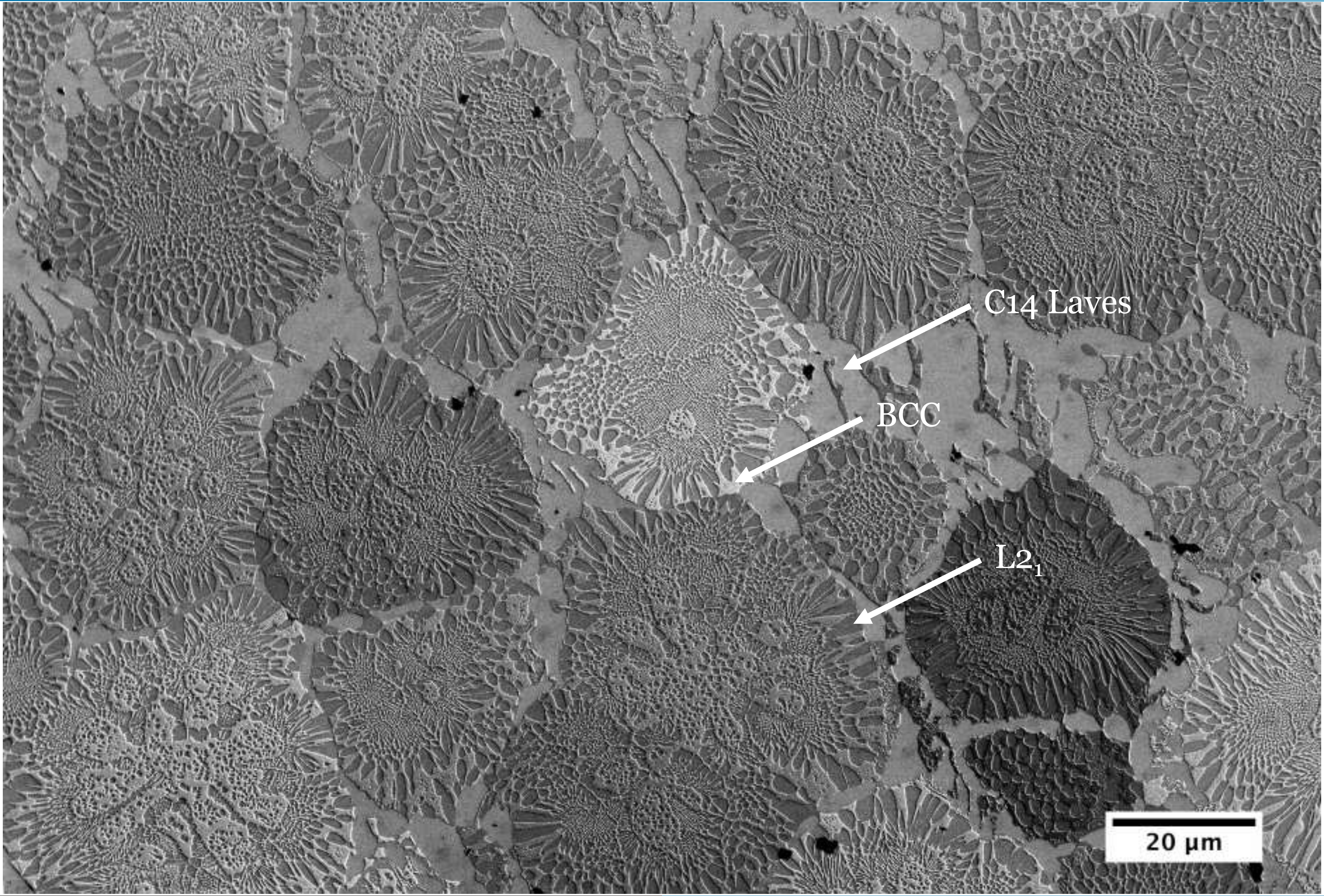


- Coupled with XRD, can identify phases in the microstructure
- Shows which elements segregate to which phases

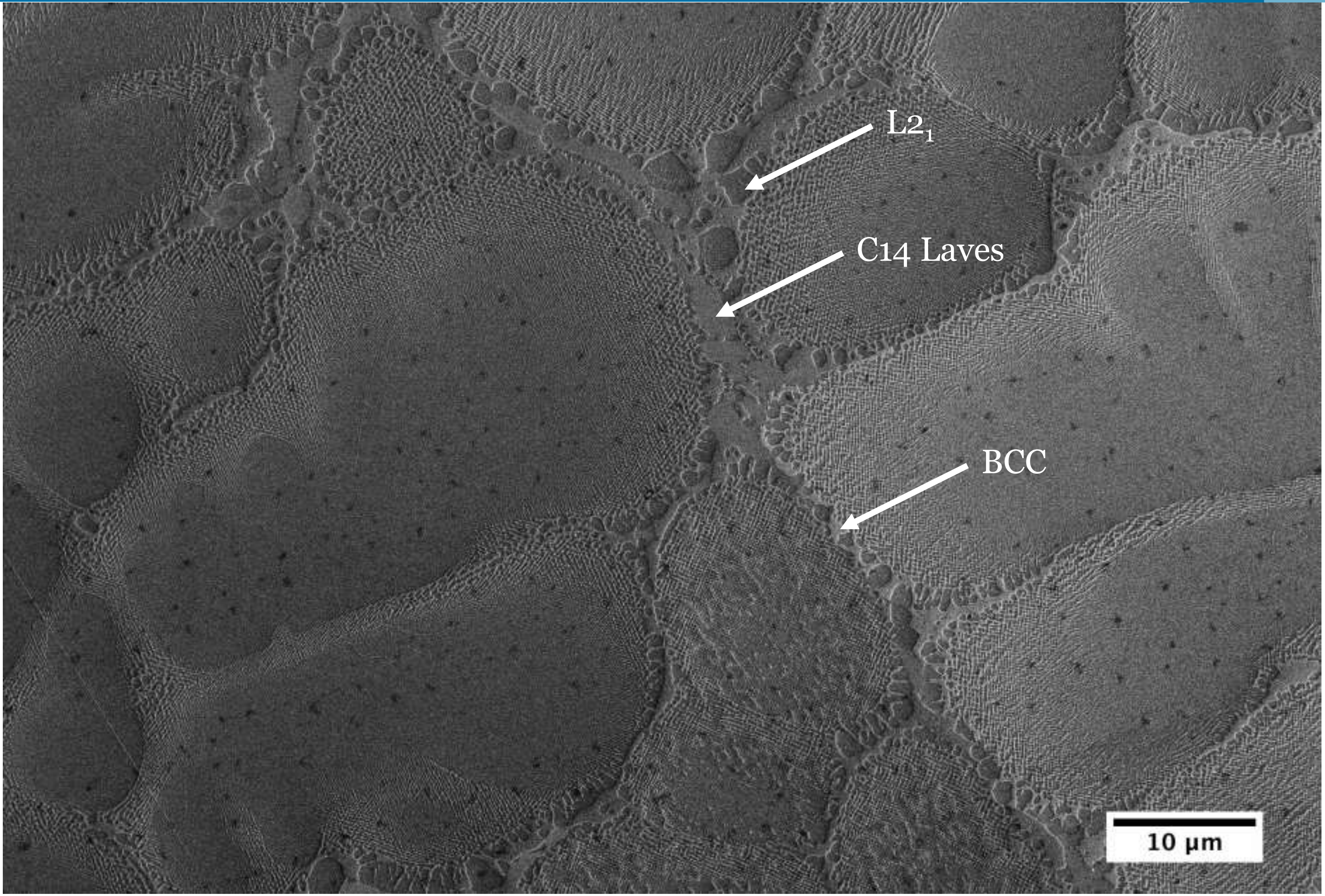
SEM Microstructures – B5 – $Al_{10}Cr_{30}Fe_{30}Ni_{20}Ti_{10}$



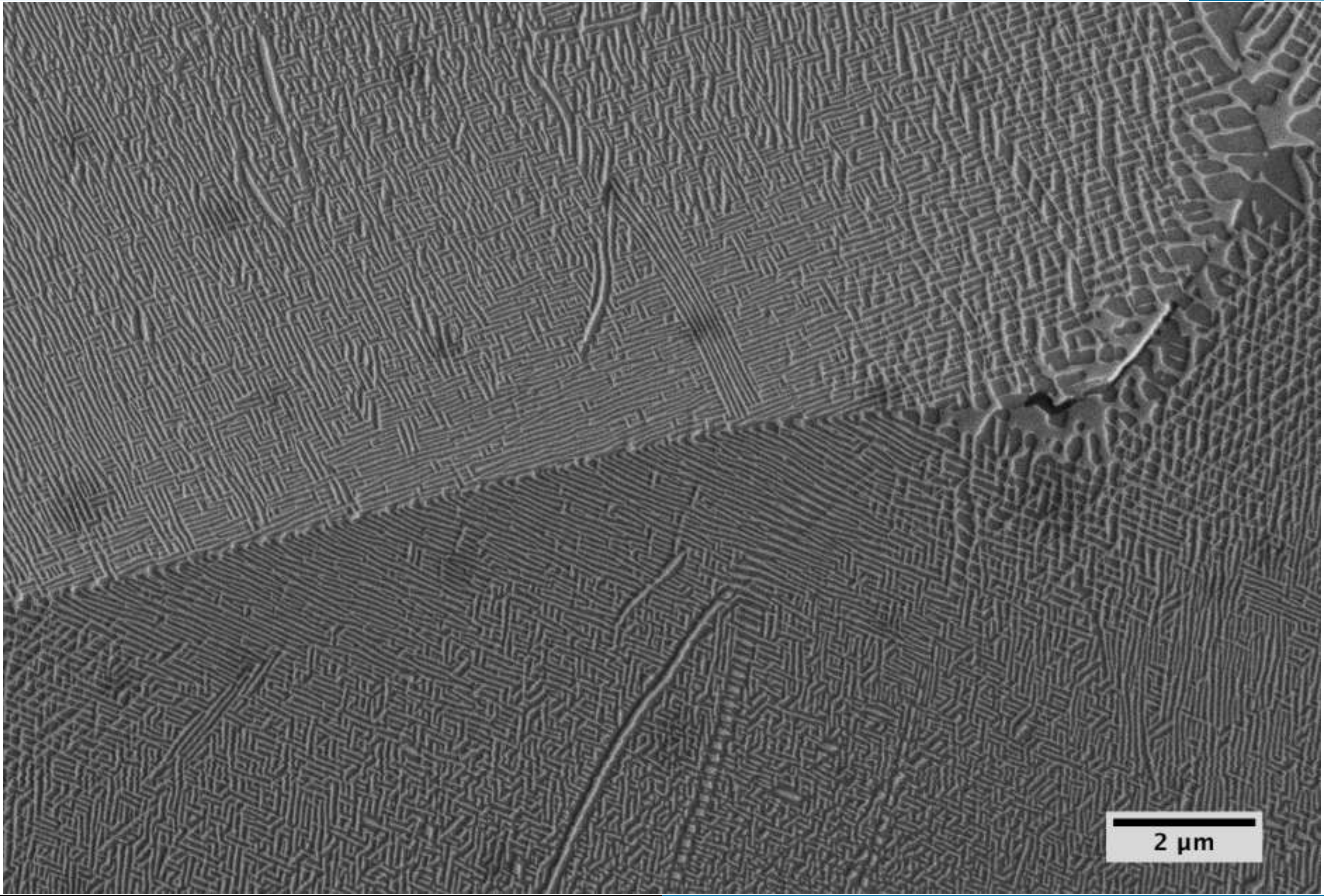
SEM Microstructures – B11 – $Al_{15}Cr_{20}Fe_{35}Ni_{15}Ti_{15}$

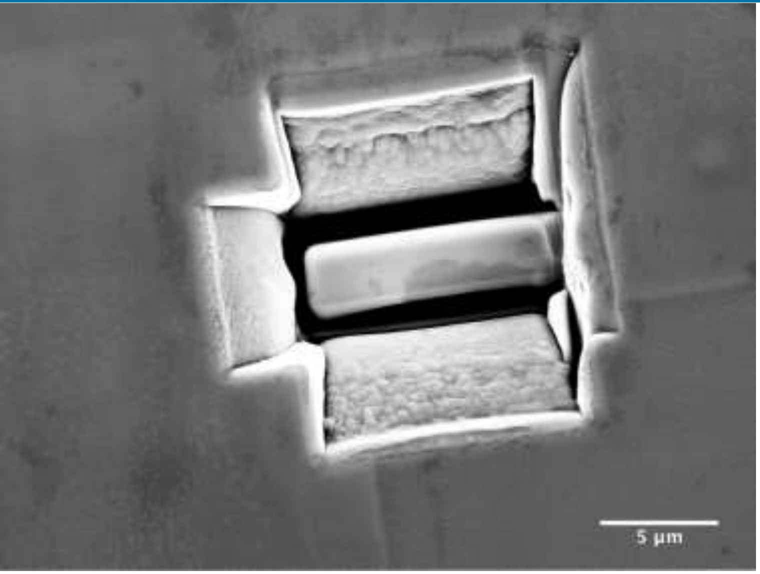


SEM Microstructures – B12 – $Al_{20}Cr_{10}Fe_{35}Ni_{20}Ti_{15}$

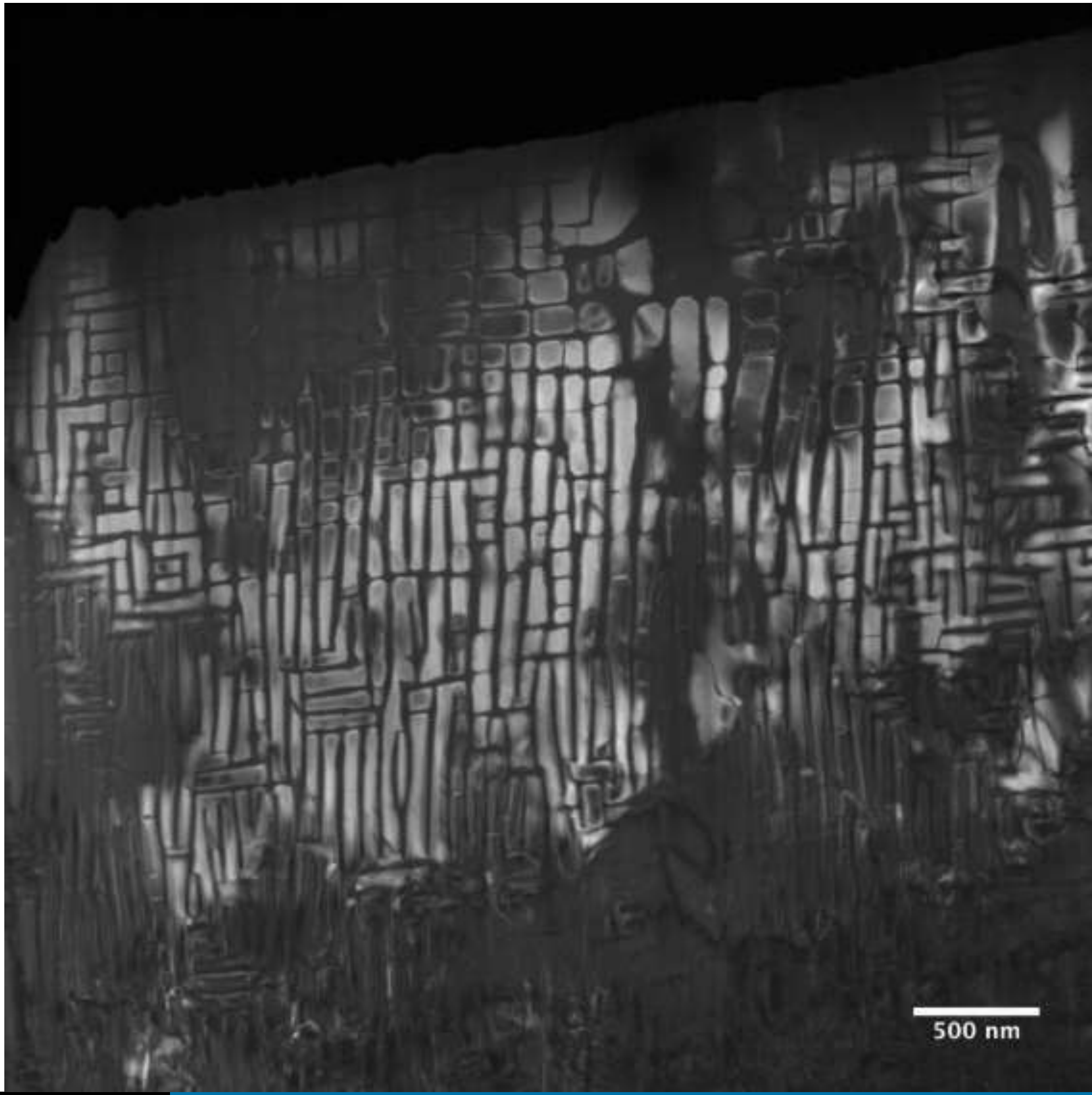


SEM Microstructures – B15 – $Al_{25}Cr_{15}Fe_{30}Ni_{20}Ti_{10}$



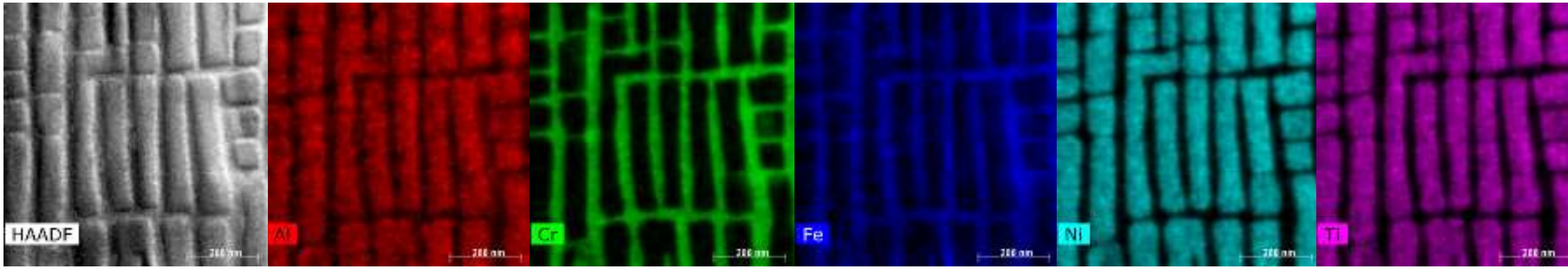


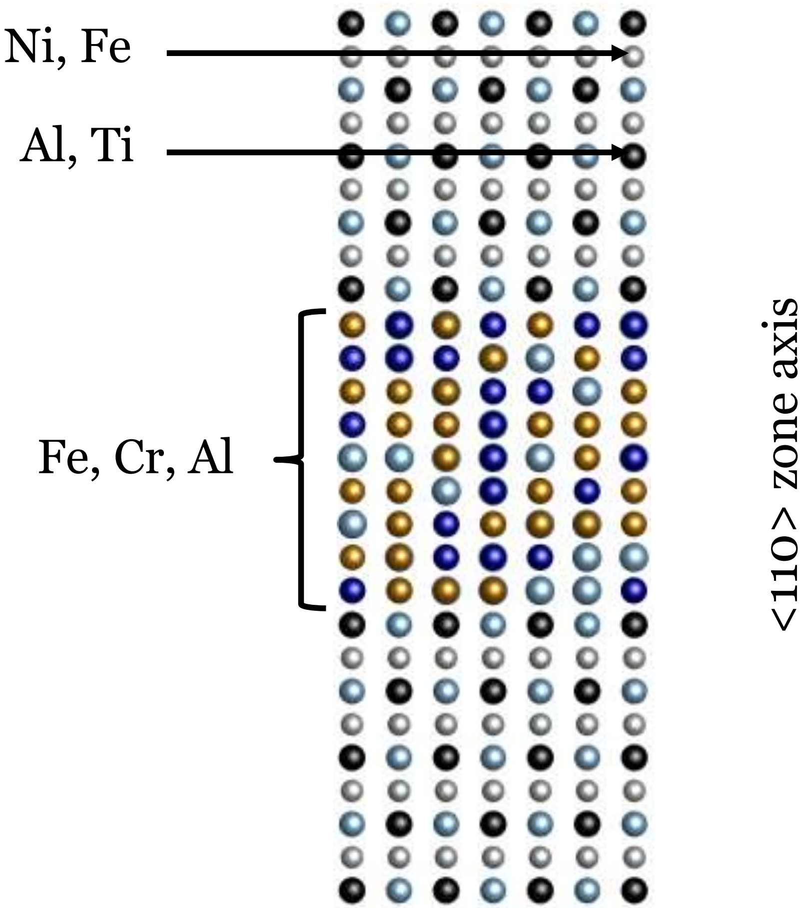
Side-view TEM lamella prepared by FIB

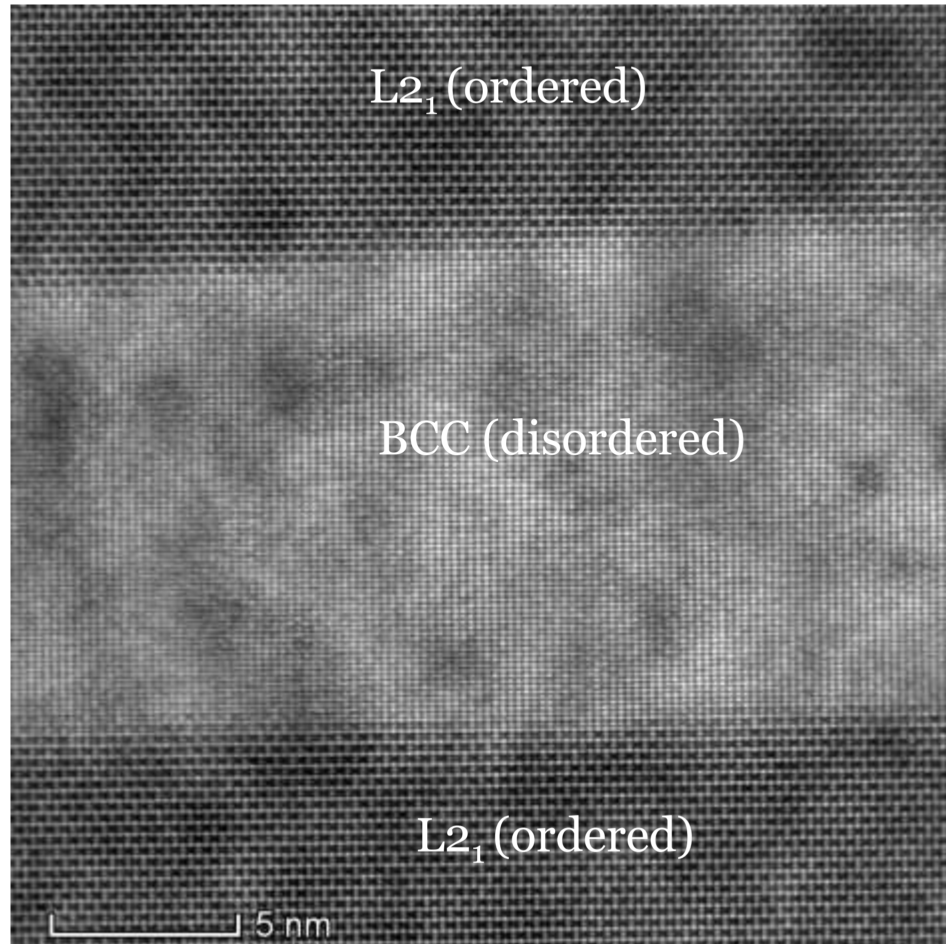


Two beam diffraction condition with <110> zone axis and $g_{111\text{-}L21}$ diffraction spot highlights $L2_1$ precipitates

STEM Analysis of B15







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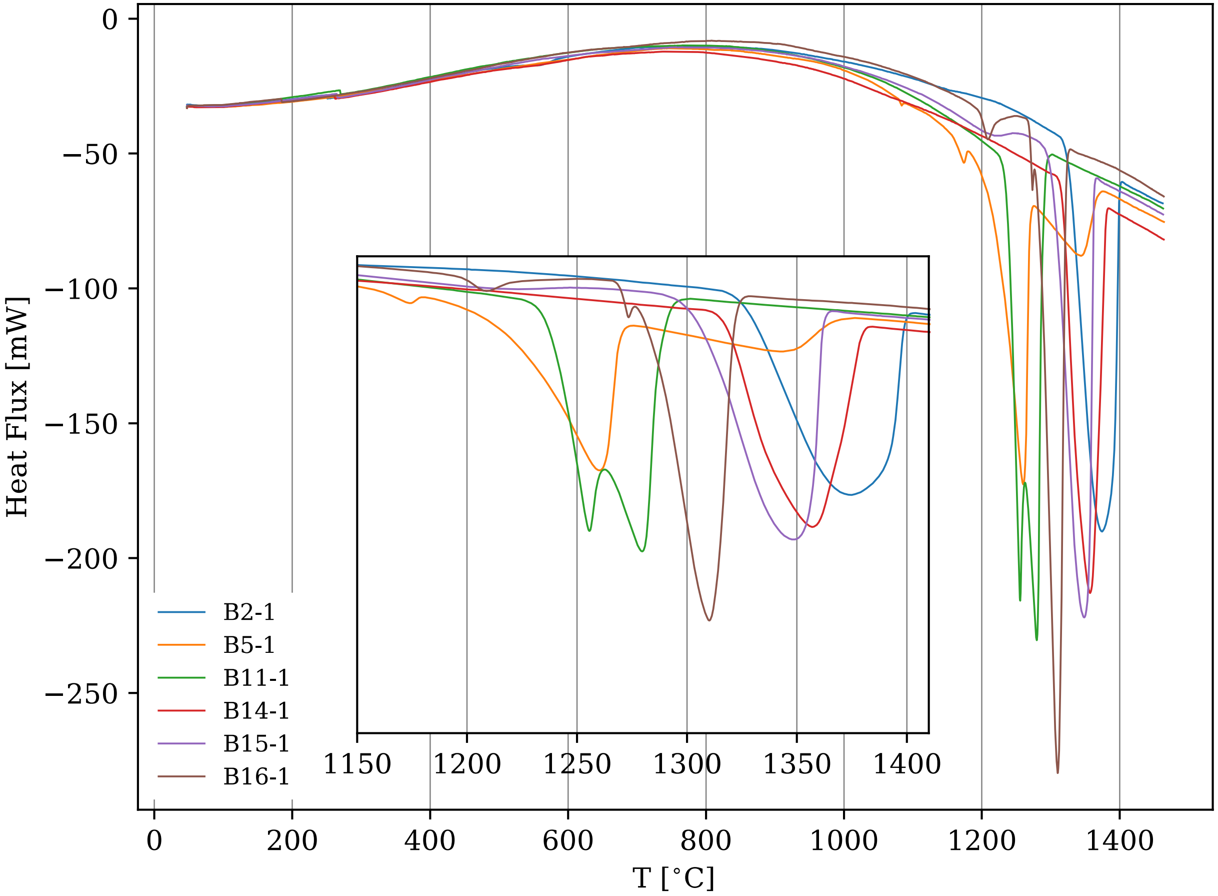
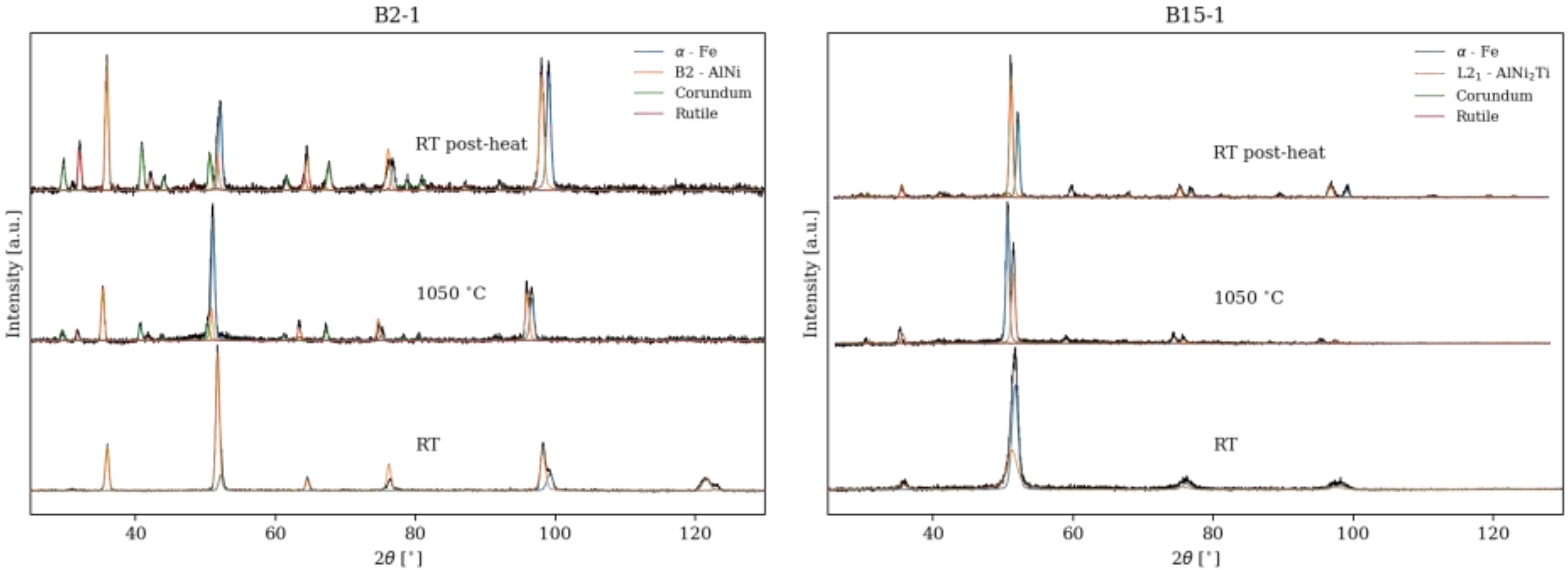
DSC Results



Sample ID	Solidus Temperature (°C)
B1-1	1271
B2-1	1327
B5-1	1220
B8-1	1182
B9-1	1196
B10-1	1180*
B11-1	1237
B12-1	1245
B14-1	1318
B15-1	1306
B16-1	1269

For most interesting materials, no observable phase changes below 900 °C

High Temperature XRD – Samples B2 and B15

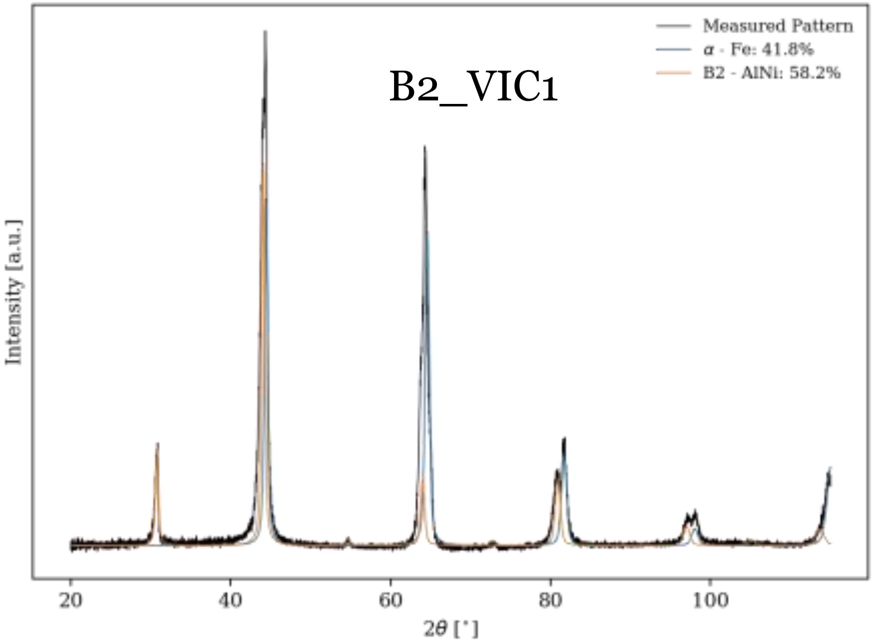


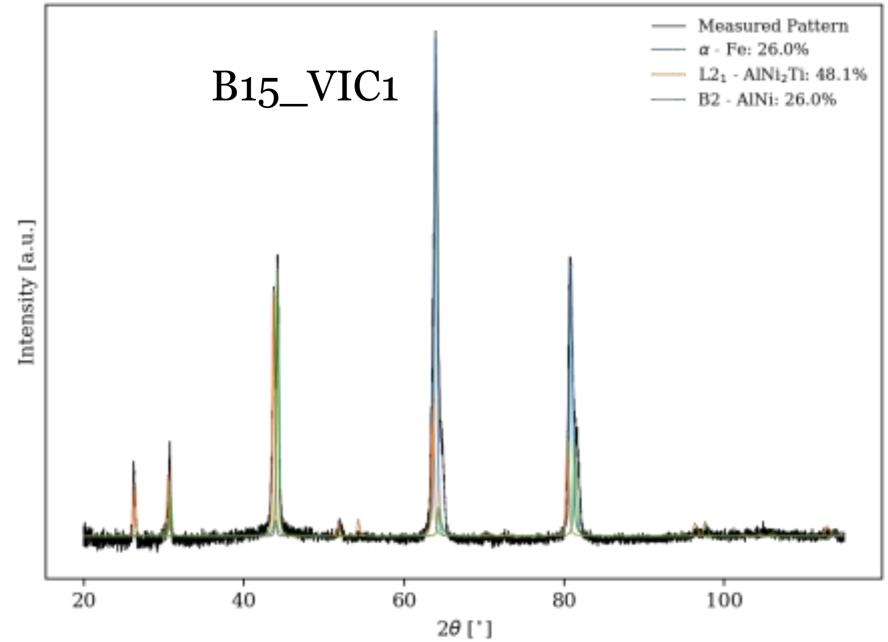
- At 1050 ºC, oxidation products TiO_2 and Al_2O_3 are observed
- Oxidation products remain on surface after cooling back to RT
- Main matched phases at RT before and after heating are BCC and B2+$L2_1$
- For sample B2, at 1050 ºC the $L2_1$ superlattice reflection is not observed, indicating that $L2_1$ has converted to B2

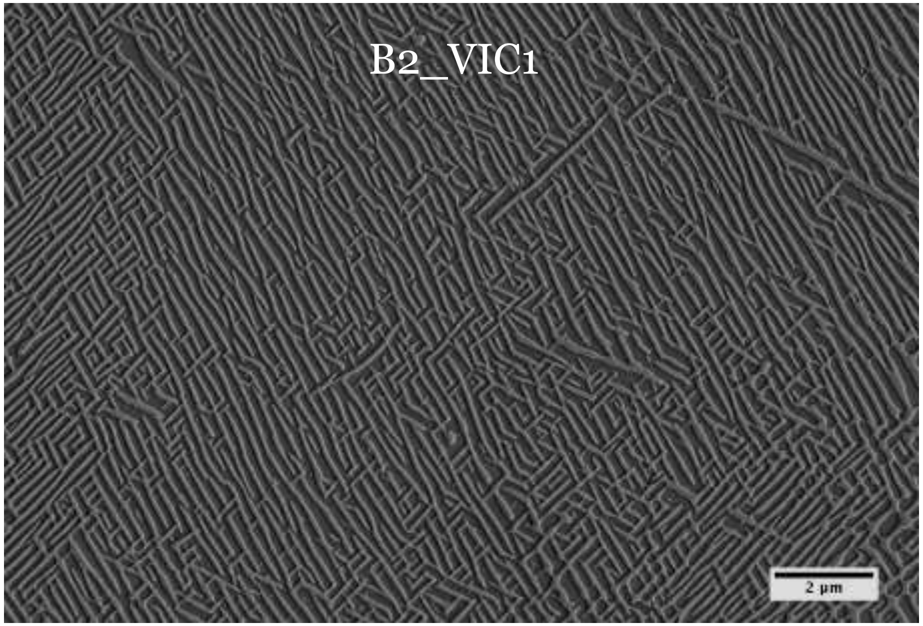
Arc Melted Results Summary

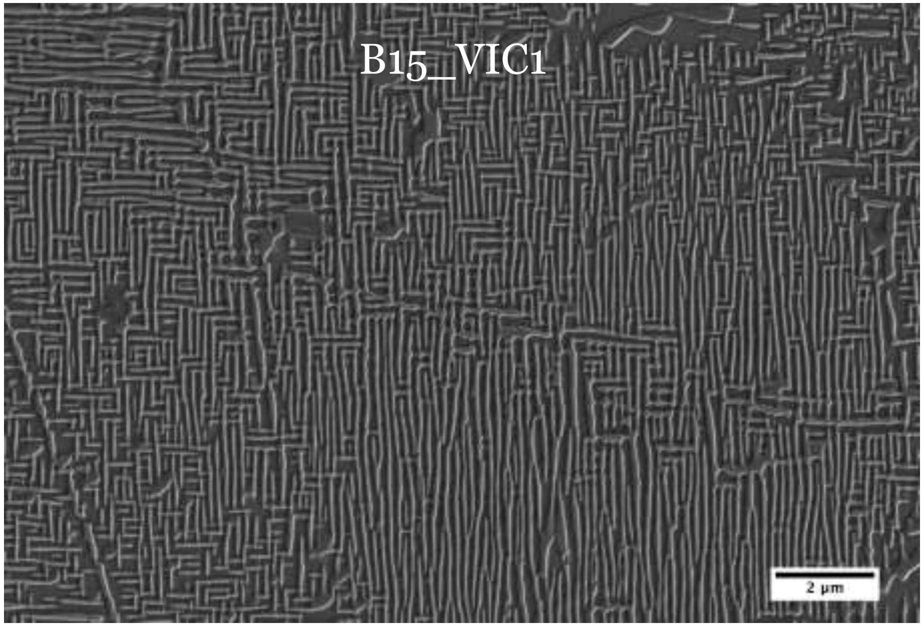
Sample ID	Composition (at%) Al	Cr	Fe	Ni	Ti	Microstructure Rating (1-5)	Density (g/cm^3) Rating (1-5)	Cost ($/kg) Rating (1-5)	Vickers Hardness (HV) Rating (1-5)	Solidus Temperature (°C) Rating (1-5)	Average (1-5)
B1	20	20	20	20	20	0.21 BCC + 0.71 ($L2_1$+B2) + 0.08 Laves 4	5.81 3	5.09 3	534 3	1271 3	3.1
B2	25	10	35	25	5	0.43 BCC + 0.57 ($L2_1$+B2) 5	6.17 2	4.92 3	416 1	1327 5	3.2
B3	35	10	10	10	35	Laves 1	4.60 5	4.56 3	726 5	- -	-
B4	35	10	10	35	10	($L2_1$+B2) 2	5.49 3	7.33 1	616 4	- -	-
B5	10	30	30	20	10	0.63 BCC + 0.37 ($L2_1$+B2) + trace Laves 4	6.73 1	4.49 3	554 3	1220 2	2.7
B6	17.5	15	17.5	15	35	0.05 ($L2_1$+B2) + 0.95 Laves 1	5.45 3	4.86 3	- -	- -	-
B7	5	15	25	20	35	0.03 BCC + 0.23 ($L2_1$+B2) + 0.74 Laves 1	6.15 2	5.14 3	- -	- -	-
B8	10	15	35	20	20	0.15 BCC + 0.37 ($L2_1$+B2) + 0.48 Laves 2	6.42 2	4.54 3	704 5	1182 1	2.6
B9	10	20	35	25	10	0.55 BCC + 0.37 ($L2_1$+B2) + 0.09 $L1_2$ 3	6.83 1	4.93 3	502 2	1196 1	2.1
B10	15	10	35	25	15	0.17 BCC + 0.69 ($L2_1$+B2) + 0.14 Laves 4	6.38 2	5.07 3	560 3	1180 1	2.5
B11	15	20	35	15	15	0.39 BCC + 0.54 ($L2_1$+B2) + 0.07 Laves 4	6.25 2	3.85 4	573 3	1237 3	3.1
B12	20	10	35	20	15	0.28 BCC + 0.66 ($L2_1$+B2) + 0.06 Laves 4	6.05 2	4.48 3	584 3	1245 3	3.1
B13	20	10	35	10	25	0.27 ($L2_1$+B2) + 0.73 Laves 1	5.64 3	3.43 4	696 5	- -	-
B14	20	20	35	20	5	0.40 BCC + 0.60 ($L2_1$+B2) 5	6.37 2	4.27 4	557 3	1318 5	3.6
B15	25	15	30	20	10	0.33 BCC + 0.67 ($L2_1$+B2) 5	5.92 3	4.56 3	612 4	1306 4	3.8
B16	25	20	35	5	15	0.30 BCC + 0.69 ($L2_1$+B2) + trace Laves 5	5.61 3	2.54 5	603 3	1269 3	4.0
B17	25	20	35	10	10	0.38 BCC + 0.62 ($L2_1$+B2) 5	5.82 3	3.10 5	646 4	- -	-
B18	30	15	35	10	10	0.46 BCC + 0.54 ($L2_1$+B2) 3	5.57 3	3.09 5	562 3	- -	-

Vacuum Induction Cast Samples – XRD and Microstructures

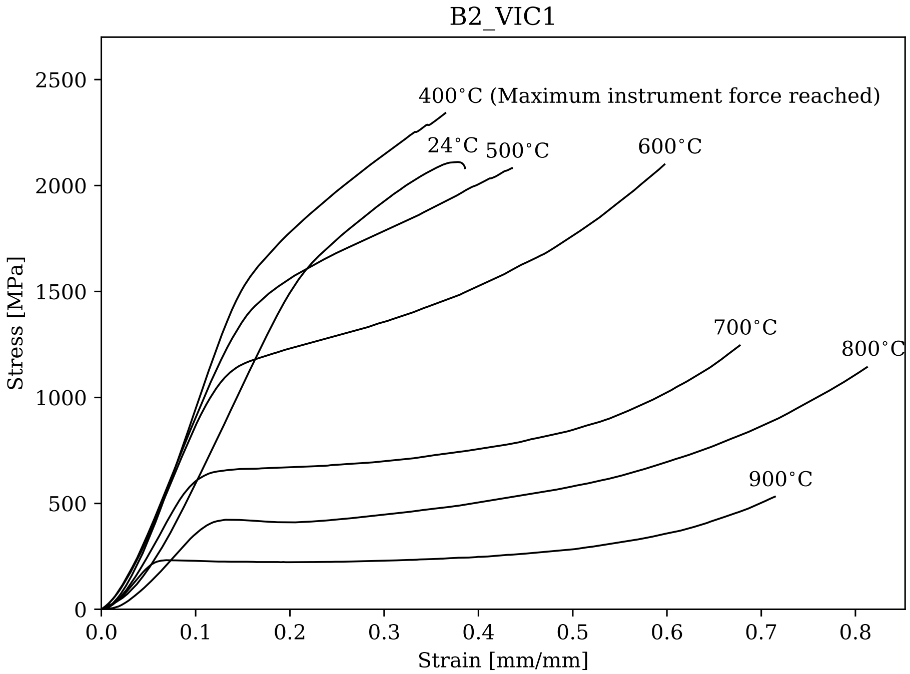


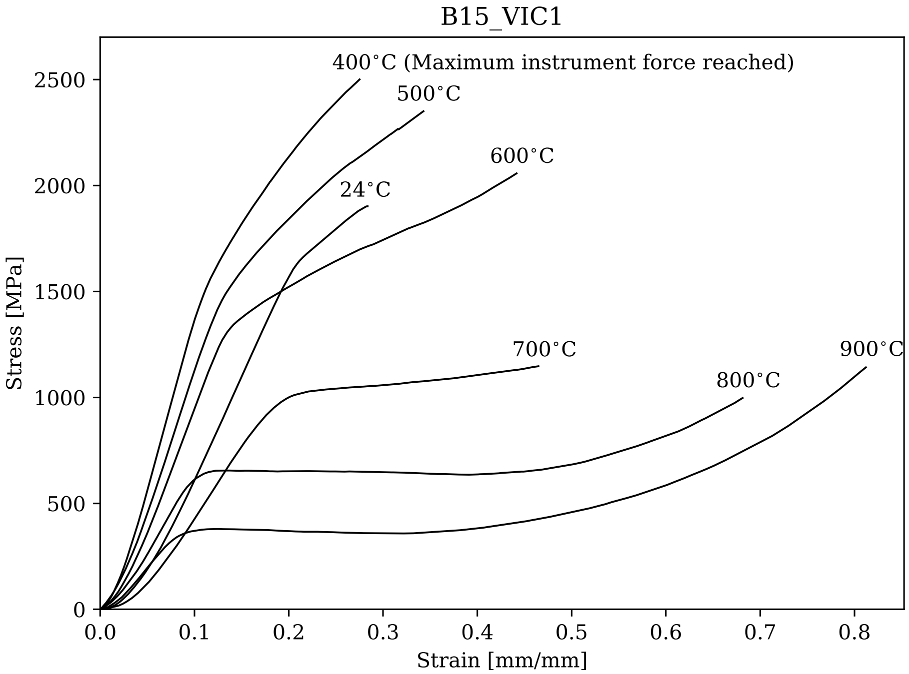




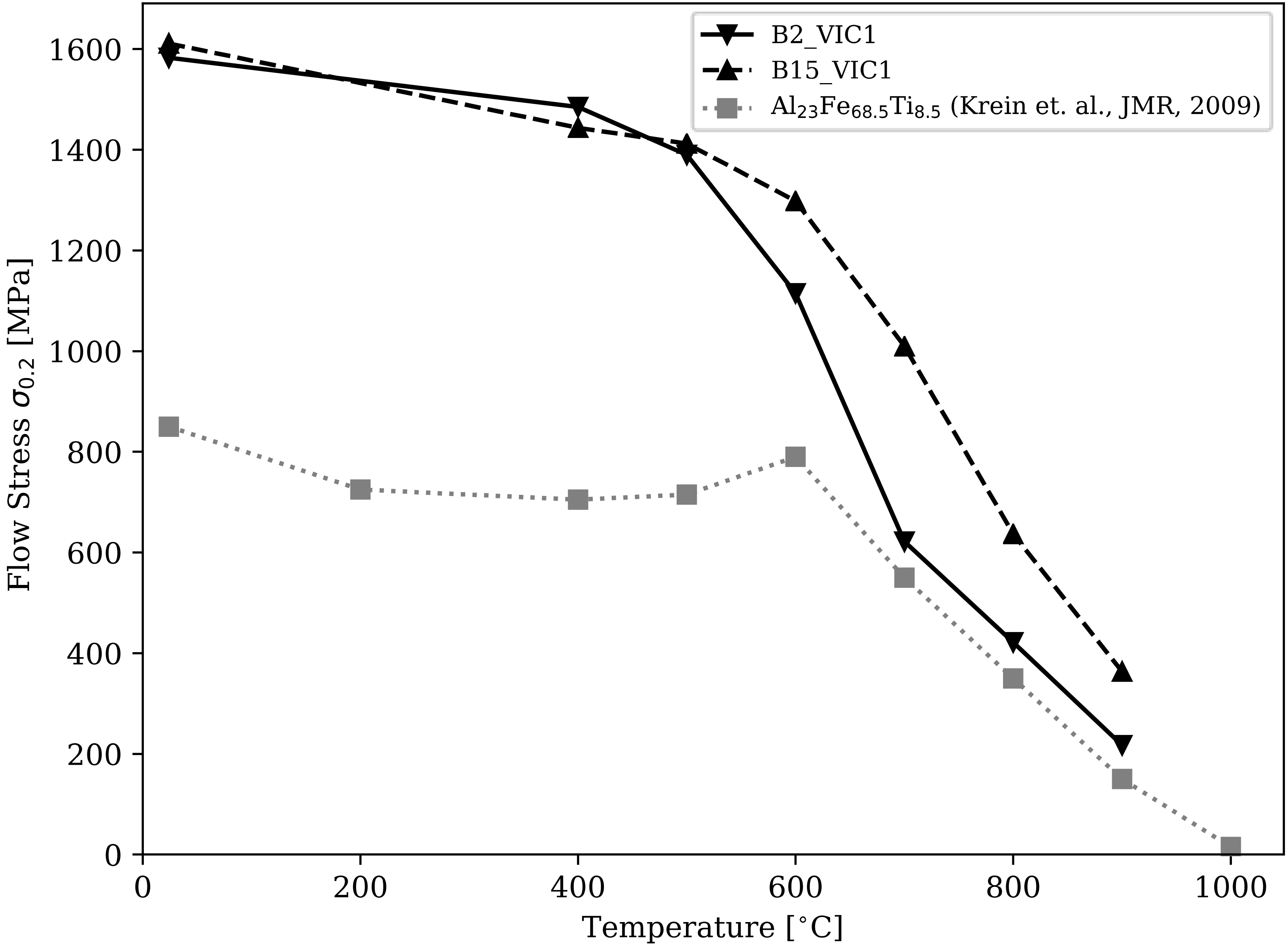


VIC Samples – High Temperature Compression Tests





- Cylindrical specimens with 5 mm height and 5 mm diameter
- Constant strain rate 0.5 mm/min
- Uniform deformation observed for all temperatures indicating homogeneous stress distribution



B2_VIC1
B15_VIC1
$Al_{23}Fe_{68.5}Ti_{8.5}$ (Krein et. al., JMR, 2009)
Flow Stress $\sigma_{0.2}$ [MPa]
0
200
400
600
800
1000
1200
1400
1600
Temperature [°C]
0
200
400
600
800
1000

Next Steps

- Elevated temperature thin film deposition and annealing
- Heat treatment of VIC samples
- Mass gain oxidation experiments
- Observe coarsening behavior by in-situ TEM
- Conduct further compression and/or tension tests at room and elevated temperatures
- Plan and carry out Creep testing

Thanks for your attention!

Questions, Comments??

Special thanks to:

- Benjamin Breitbach (XRD)
- Dr. Frank Stein (DSC and discussions)
- Dennis Klapproth (Sample synthesis)
- Thorsten Meiners (TEM)
- Herbert Faul (Mechanical Testing)

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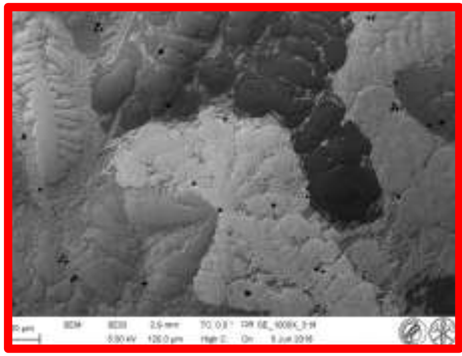
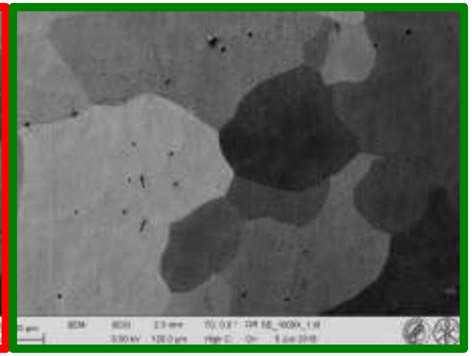
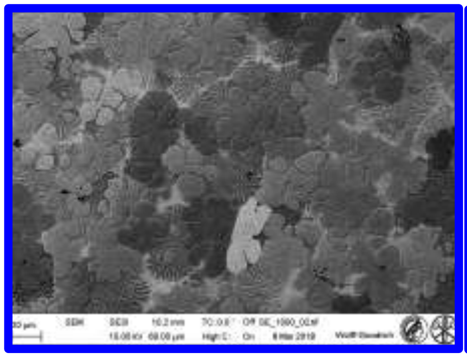
SEM Microstructures – Low Mag Summary



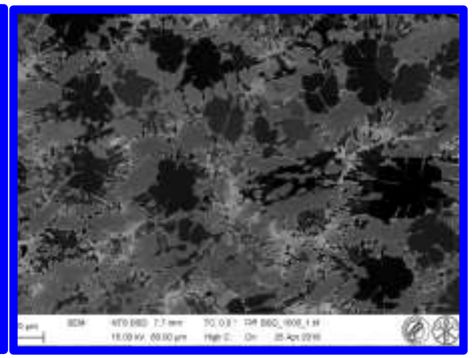
(a) B1-1



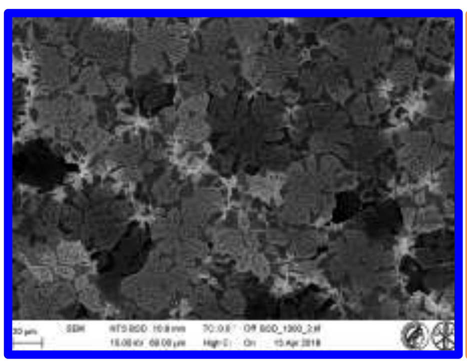
(b) B2-1



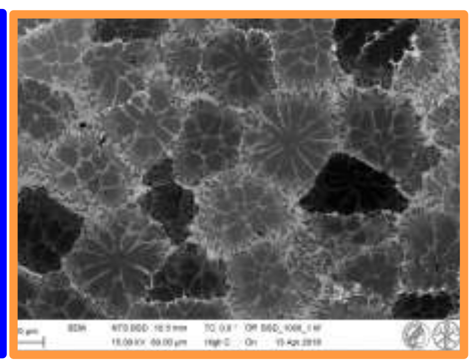
(c) B5-1



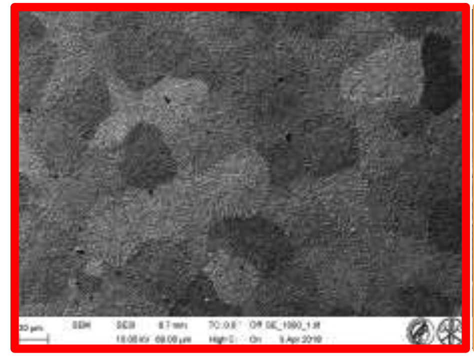
(d) B8-1



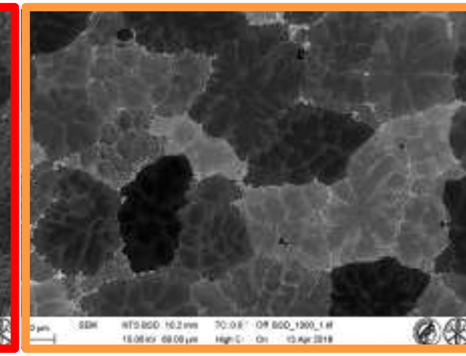
(e) B9-1



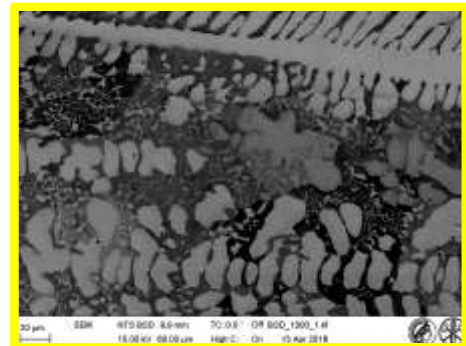
(f) B10-1



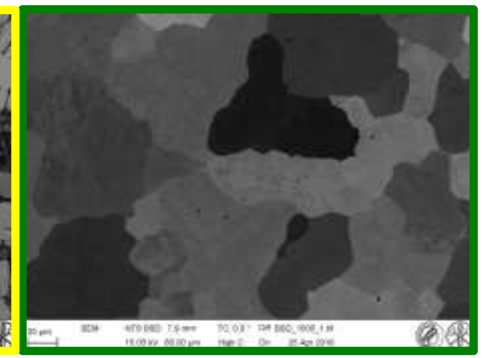
(g) B11-1



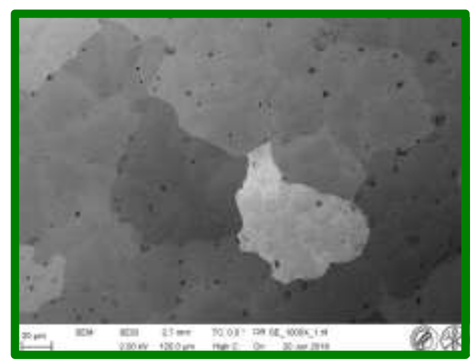
(h) B12-1



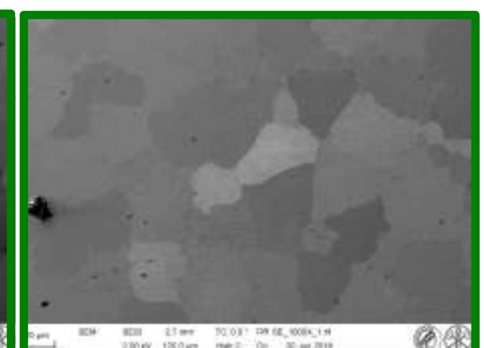
(i) B13-1



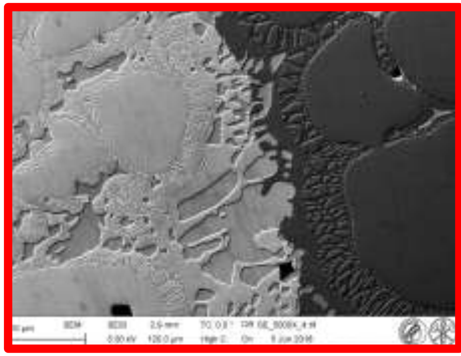
(j) B14-1



(k) B15-1



(l) B16-1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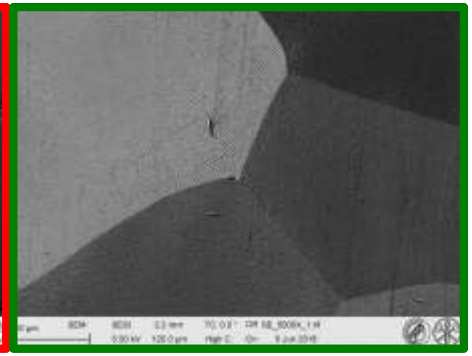
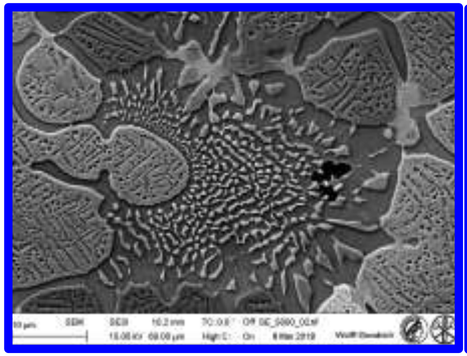
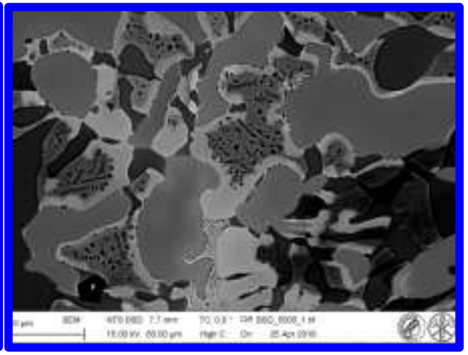
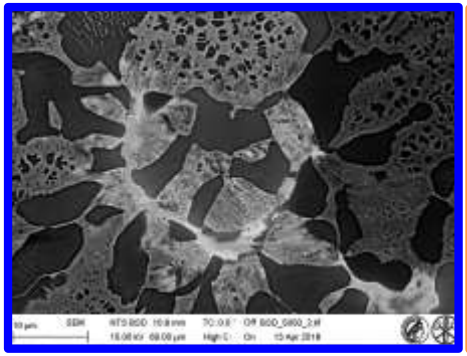

(b) B2-1


(c) B5-1


(d) B8-1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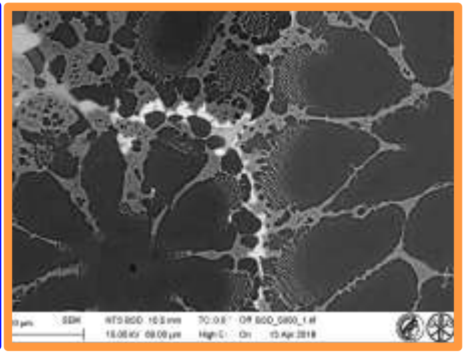
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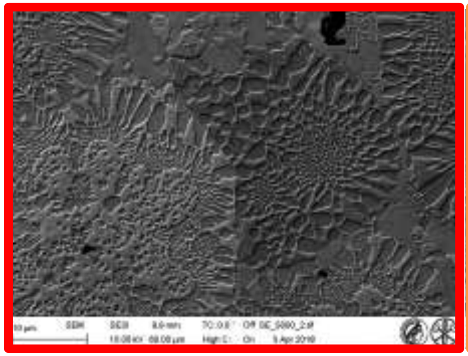
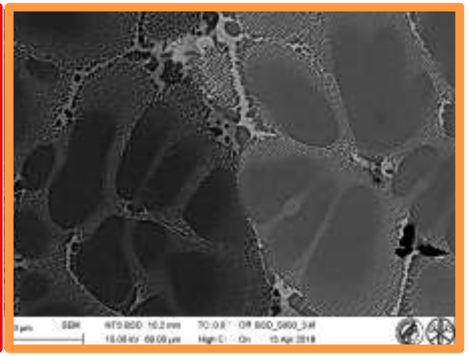

(g) B11-1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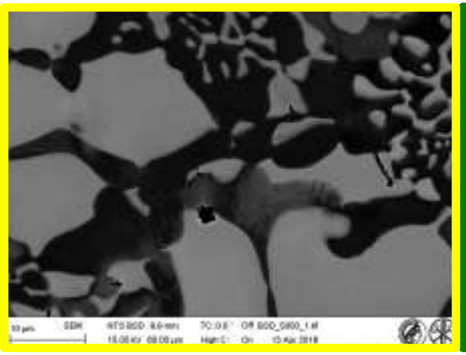
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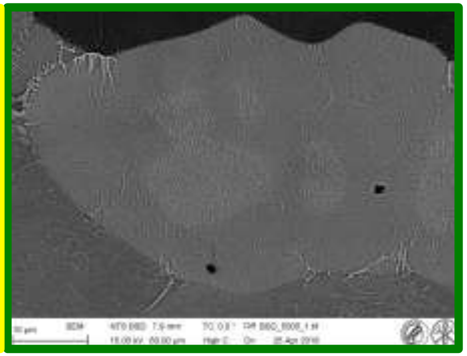
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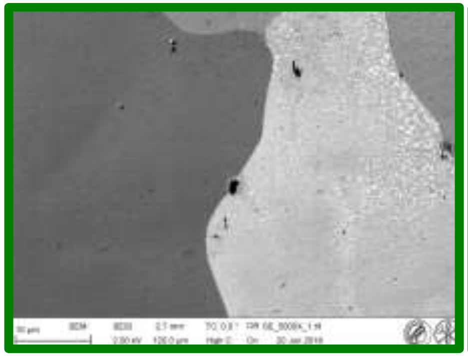
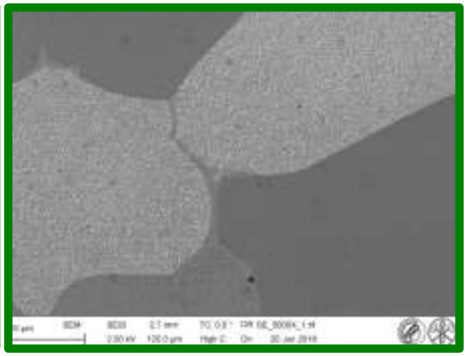
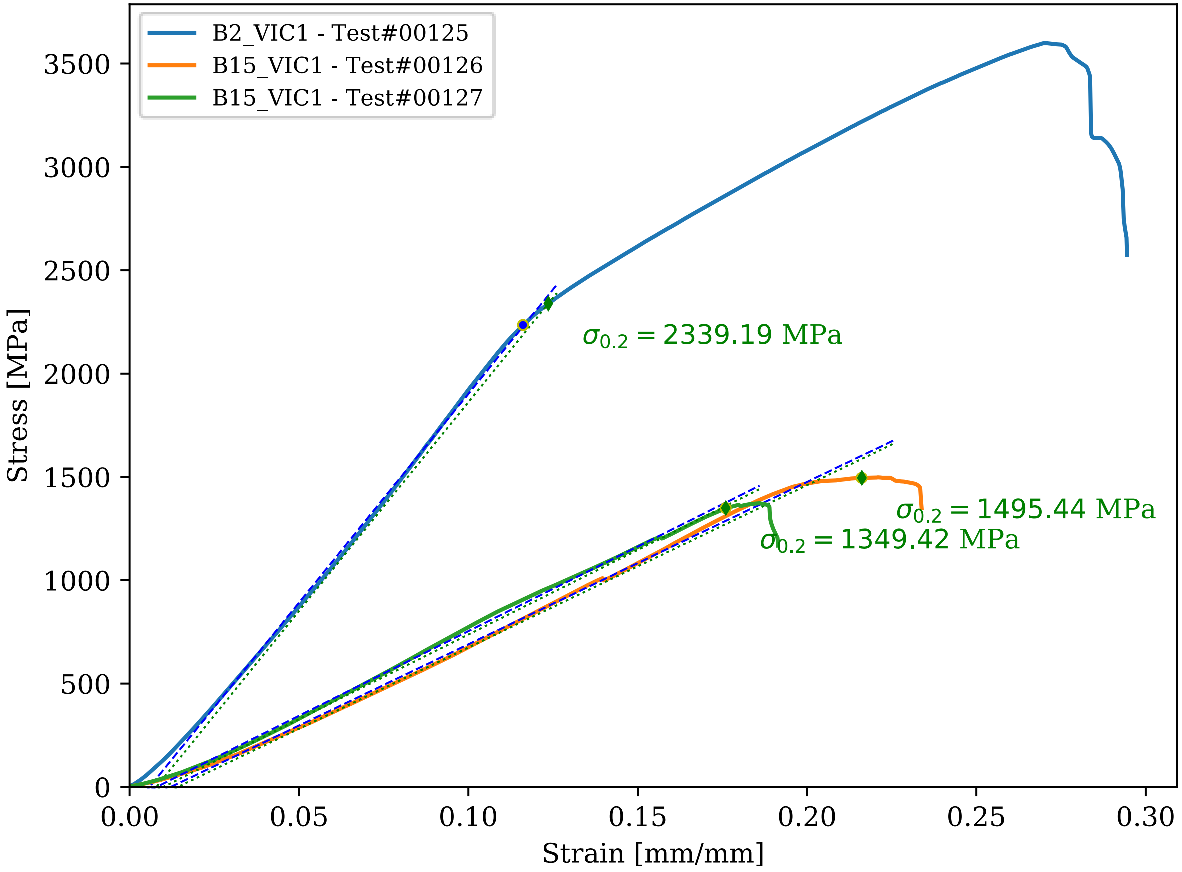

(k) B15-1


(l) B16-1



B2_VIC1 - Test#00125
B15_VIC1 - Test#00126
B15_VIC1 - Test#00127
$\sigma_{0.2} = 2339.19$ MPa
$\sigma_{0.2} = 1495.44$ MPa
$\sigma_{0.2} = 1349.42$ MPa
Stress [MPa]
Strain [mm/mm]
0
500
1000
1500
2000
2500
3000
3500
0.00
0.05
0.10
0.15
0.20
0.25
0.30