Development of Precipitation Strengthened CCAs in the AlCrFeNiTi System for High Temperature Structural Applications

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CCHEA

DFG

Rapid Screening

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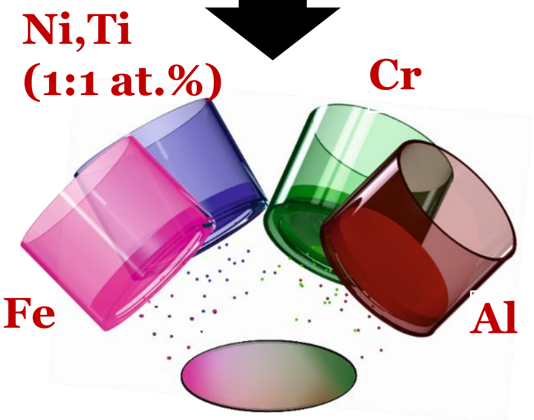


Magnetron Sputtered Thin Film Libraries

Bulk Synthesis

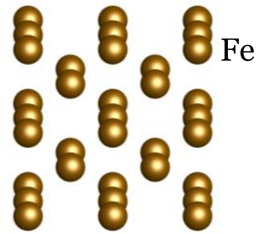
Characterization and Testing

Inspiration: Fe-base Superalloys
Goal: A2 matrix with B2 and/or $L2_1$ precipitates

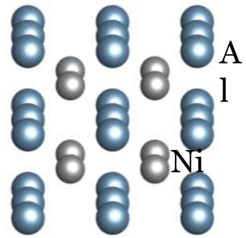
Al - Cr - Fe - Ni - Ti

Design Guidelines:

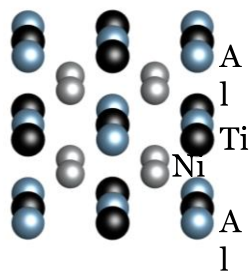
- Cheap: Maximize Fe
- Lightweight: Al, Ti Additions
- Creep Resistant: Al, Ni, Ti for $L2_1$
- Oxidation Resistant: Al, Cr
- Want large sampling area



BCC (e.g. α–Fe)

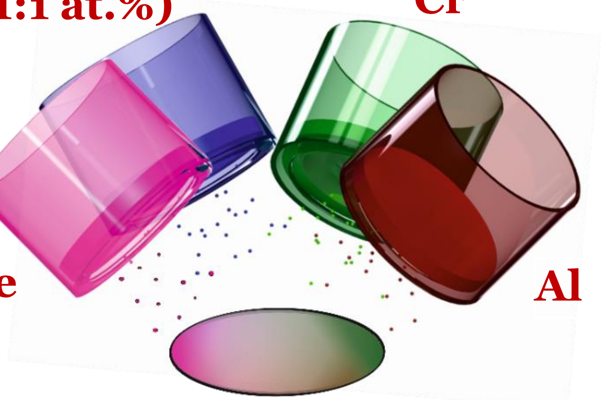


B2 (e.g. NiAl)



$L2_1$ (e.g. Ni_2TiAl)
a.k.a. Heusler Phase

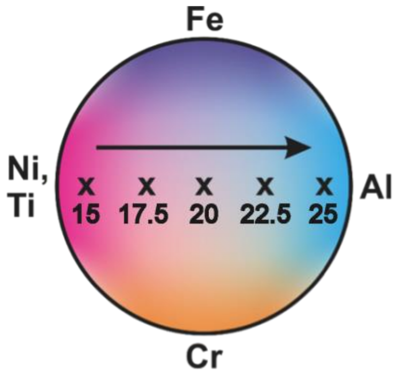
Thin Film Results



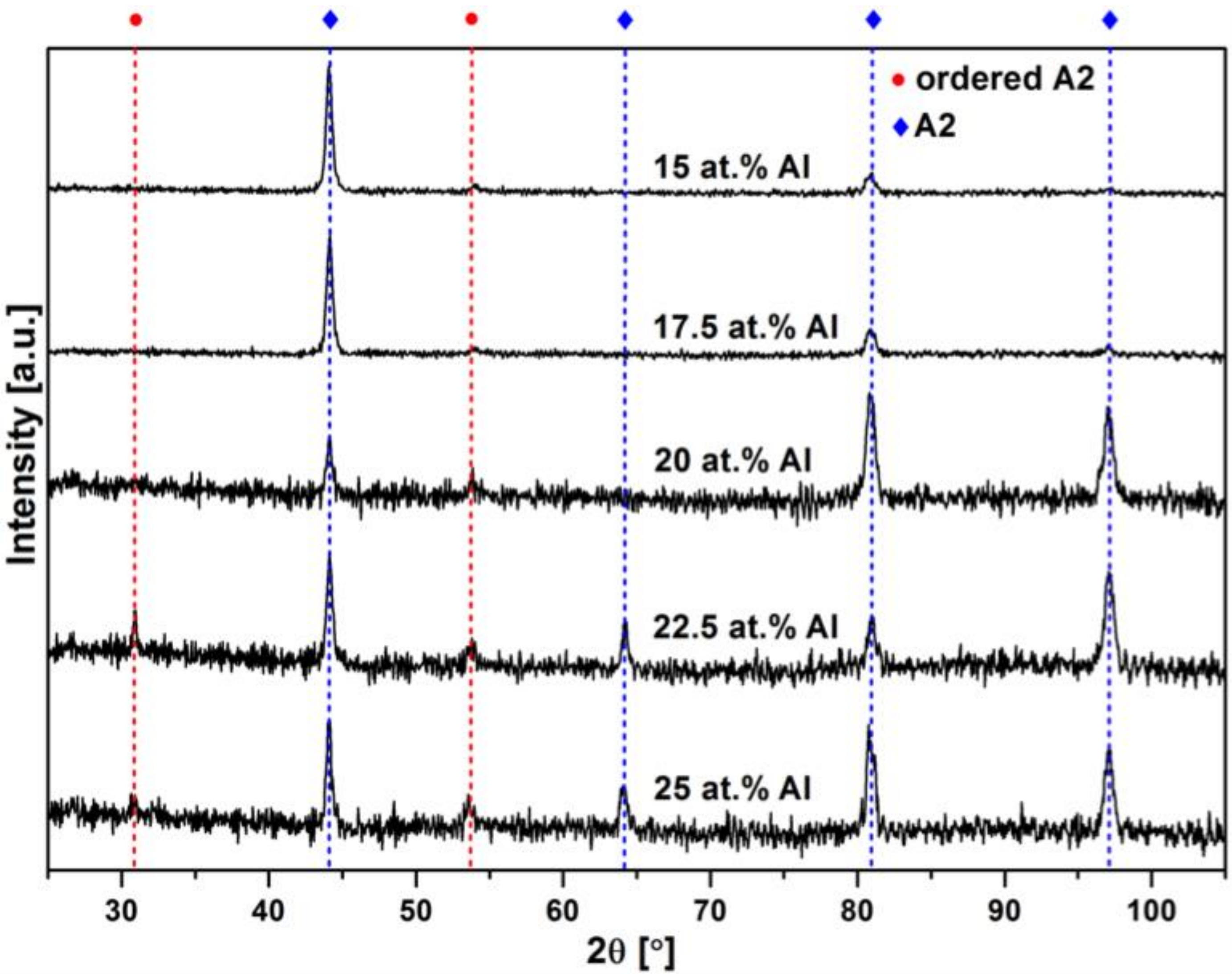
Film synthesized at 600 °C

Substrate: Si <100> w/ SiN buffer-layer

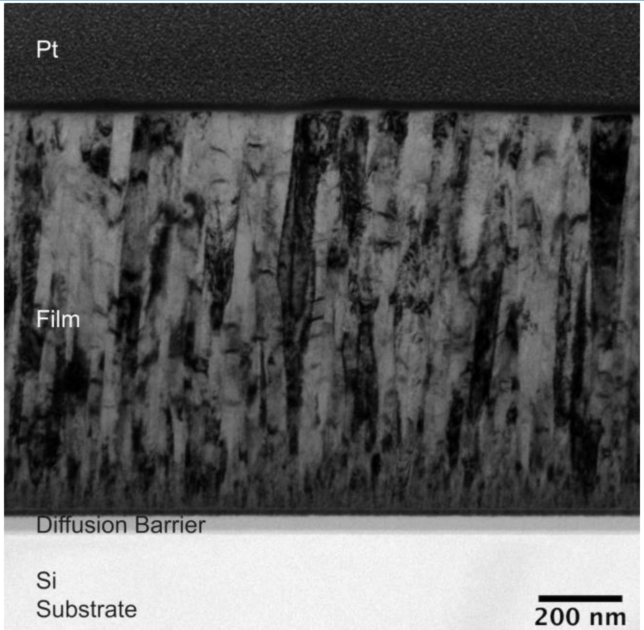
Characterized along Al concentration gradient



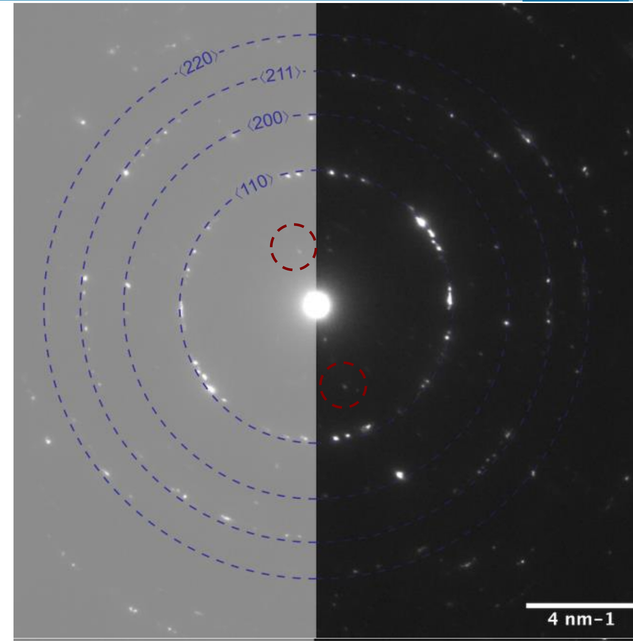
$Al_{15-25}Cr_{20}Fe_{35}(NiTi)_{45-Al\ at.\%}$

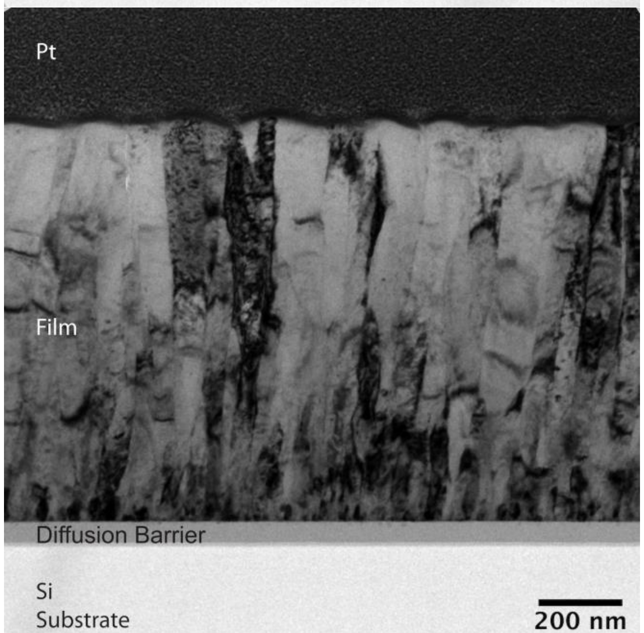


Thin Film Phase Formation

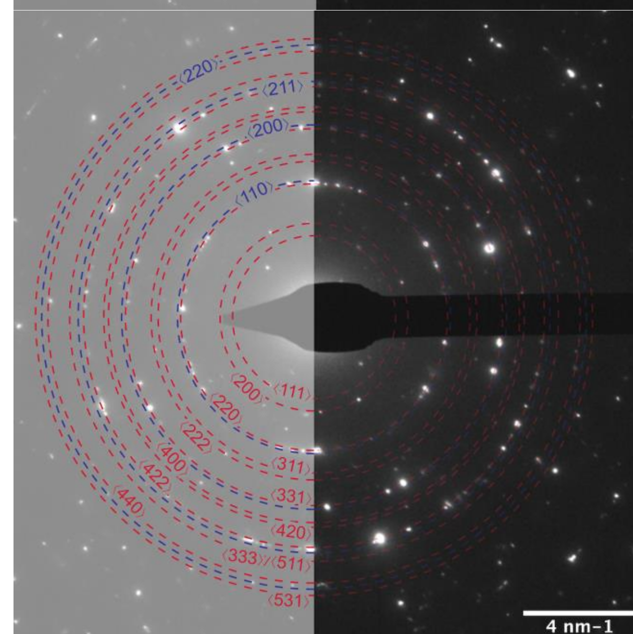


15% Al





25% Al



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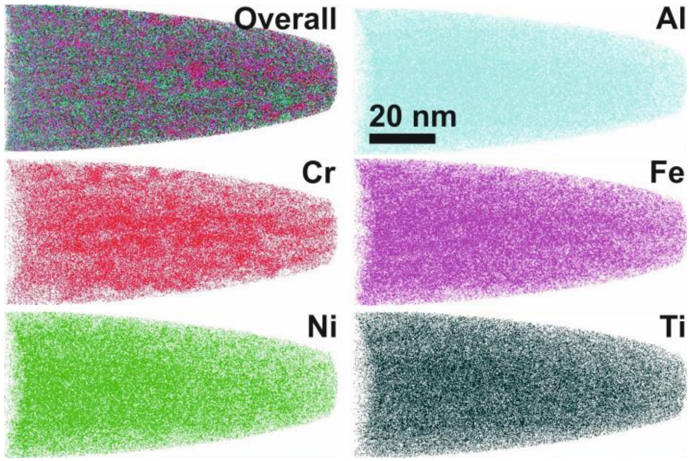
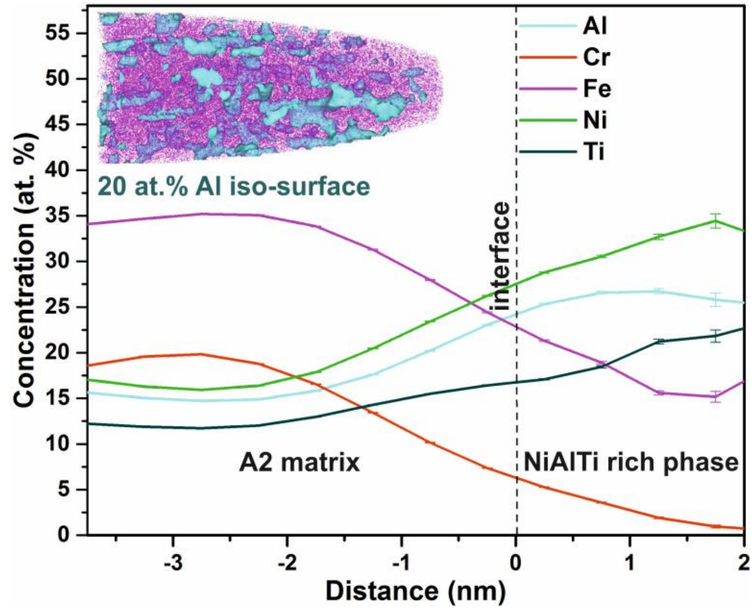
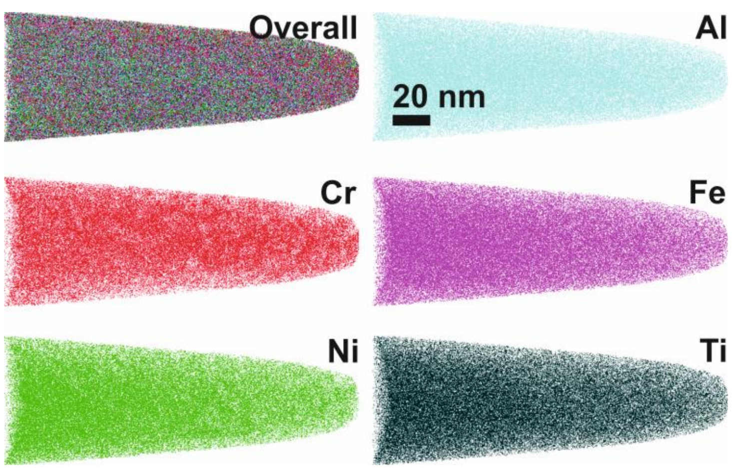
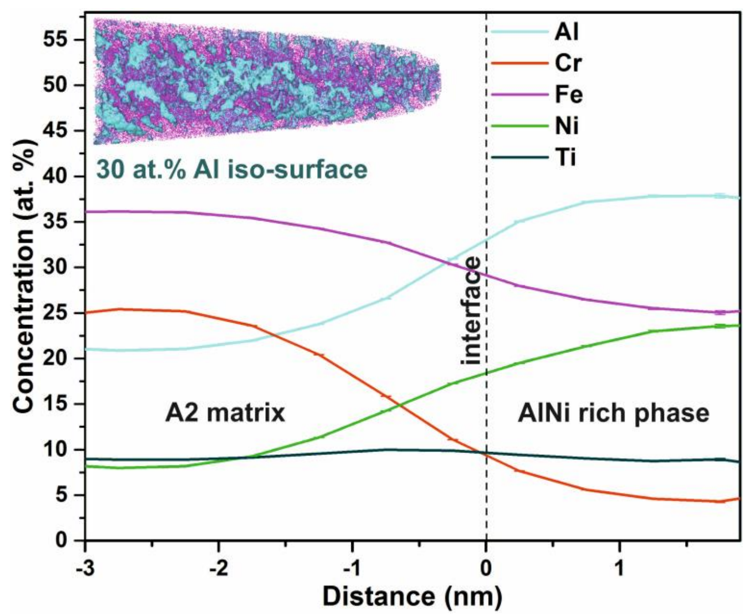


15% Al

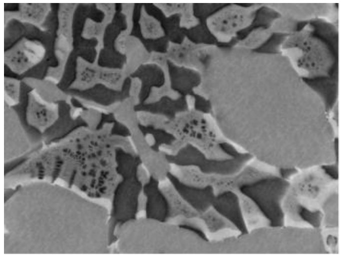
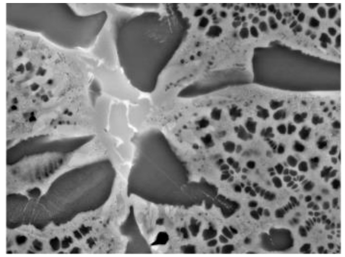
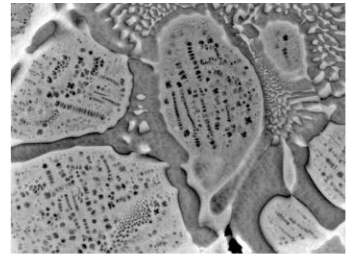
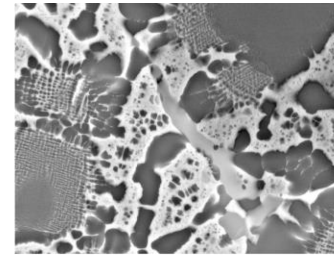
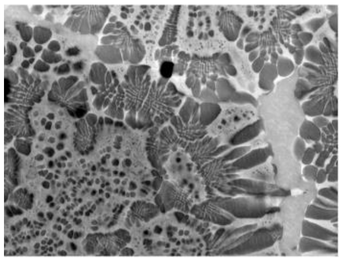
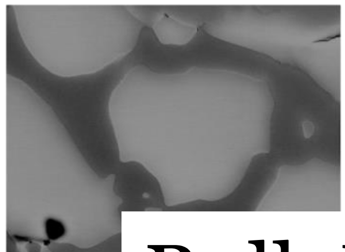
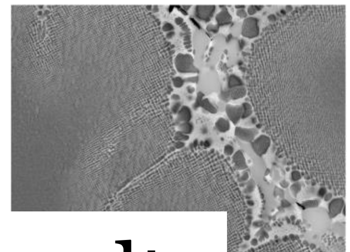
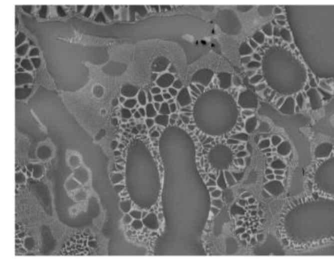
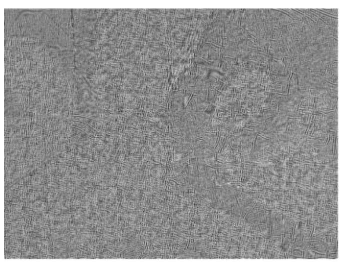
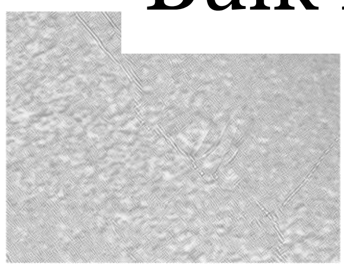
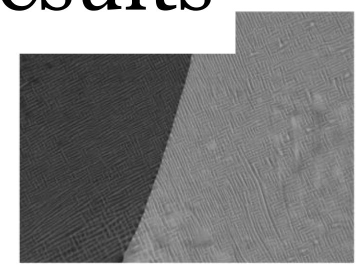
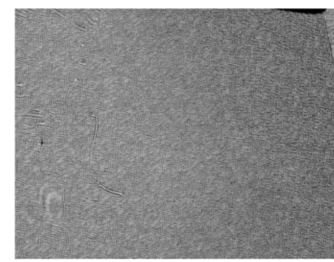
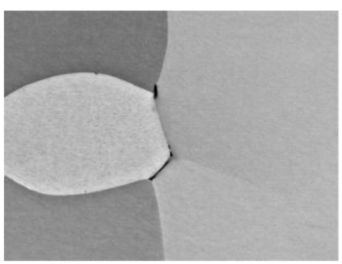
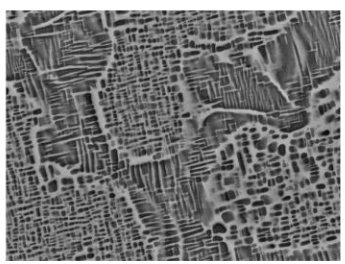
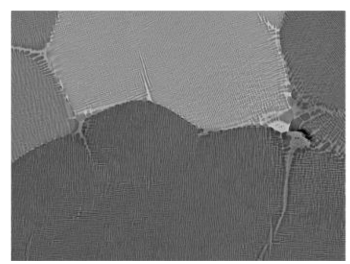
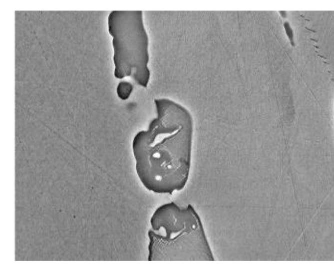




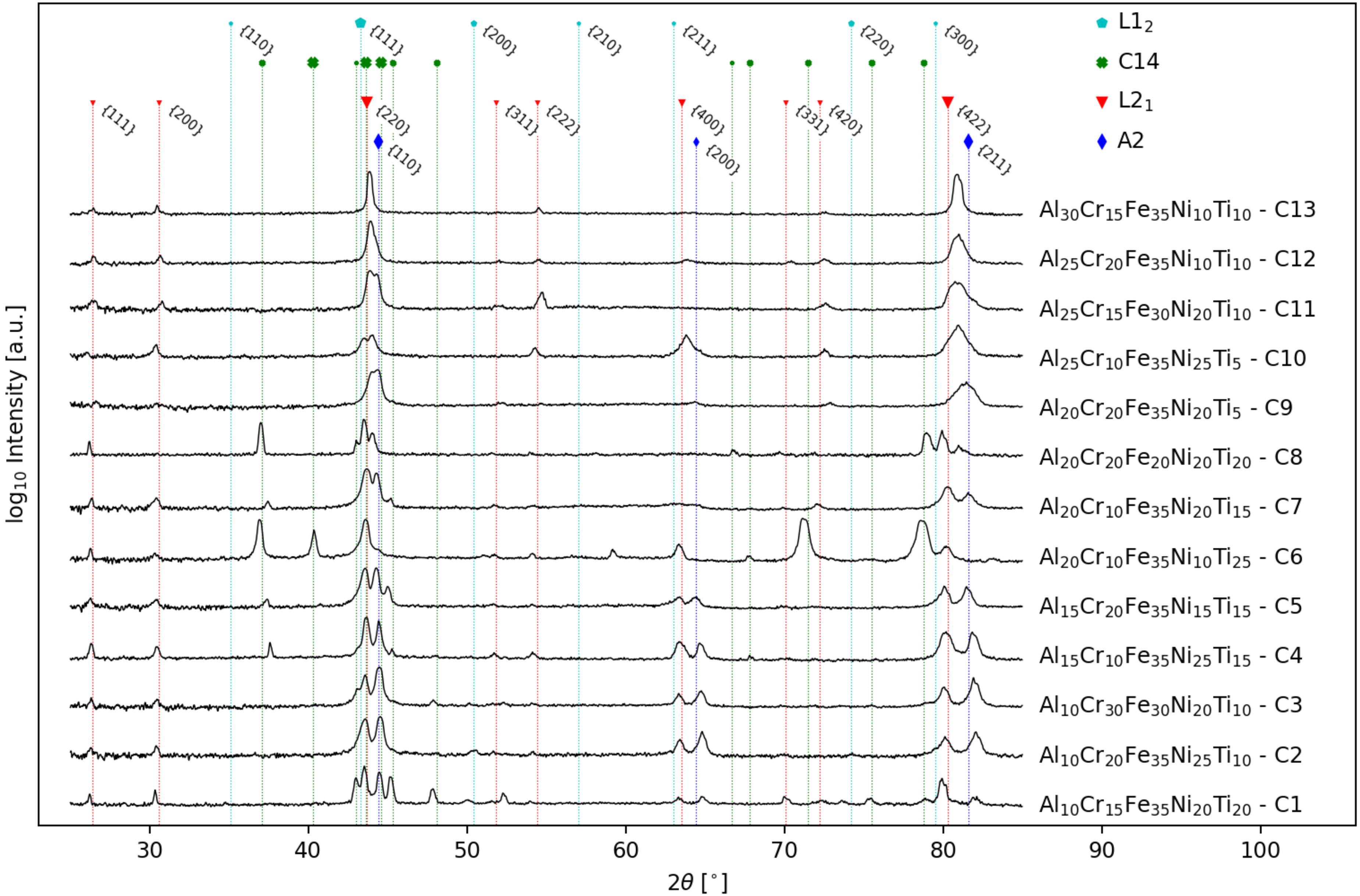
25% Al



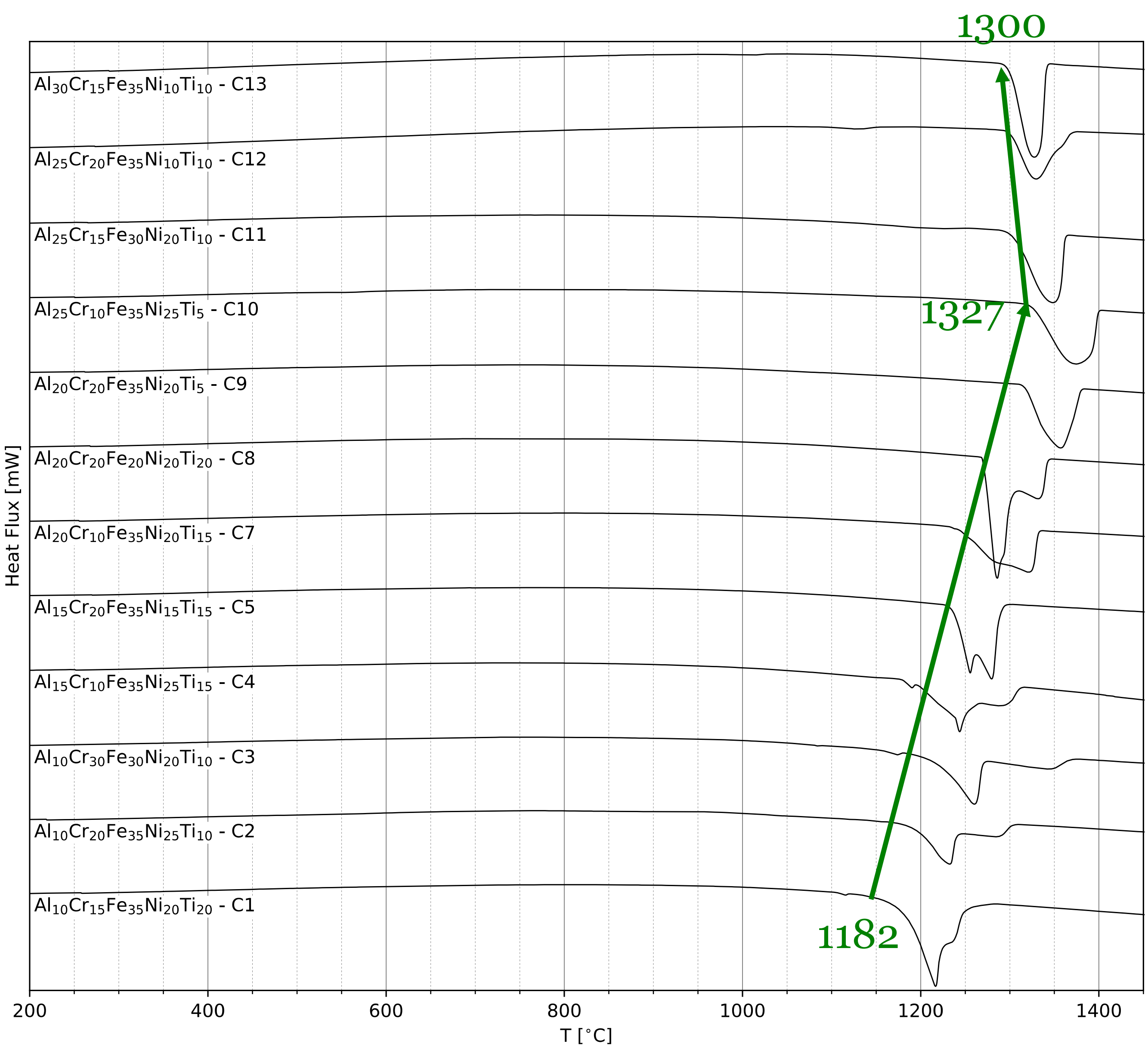
Bulk Results

XRD Results – Arc Melted Samples



DSC Results – Effect of Aluminum

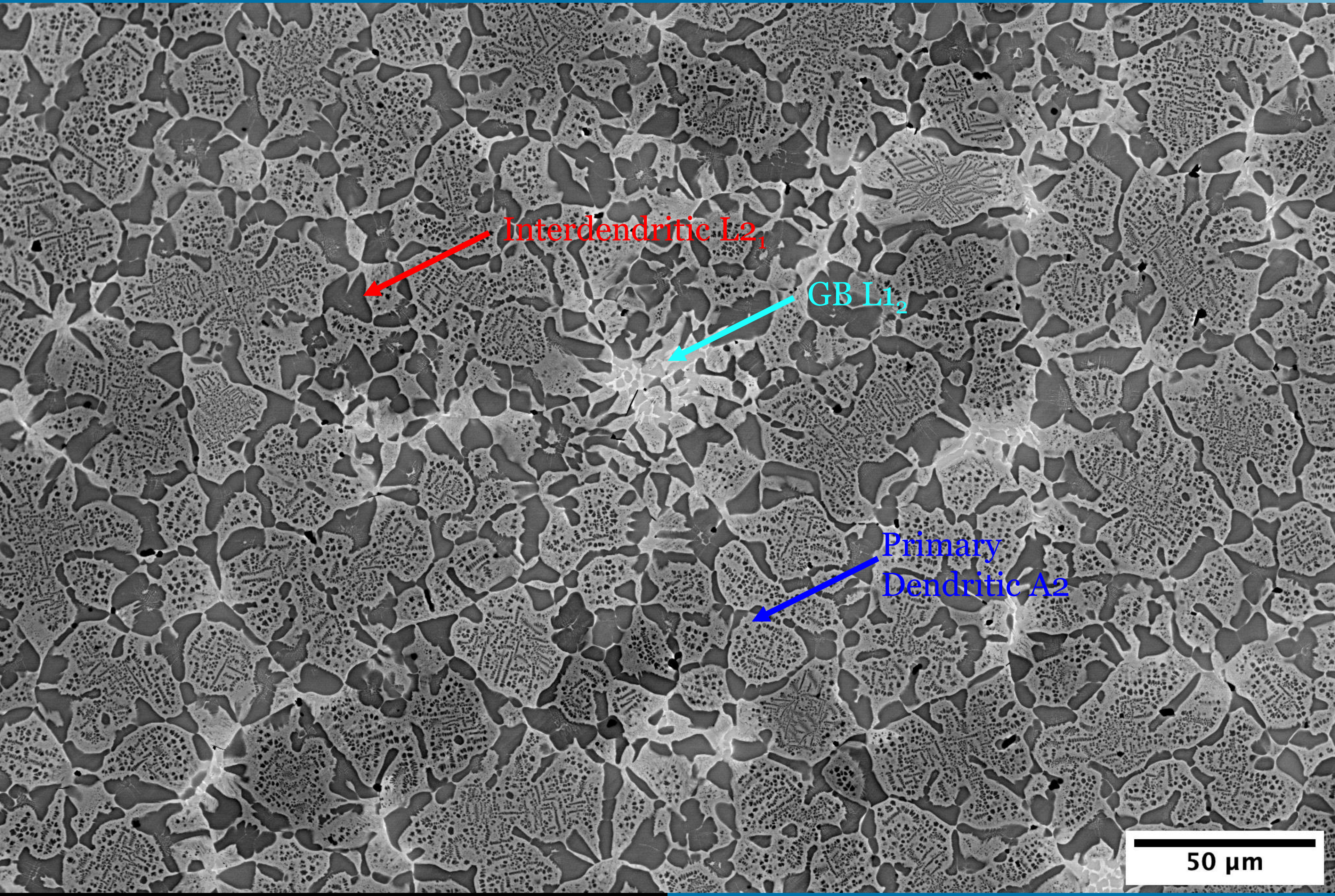


No observable phase changes below 900 °C

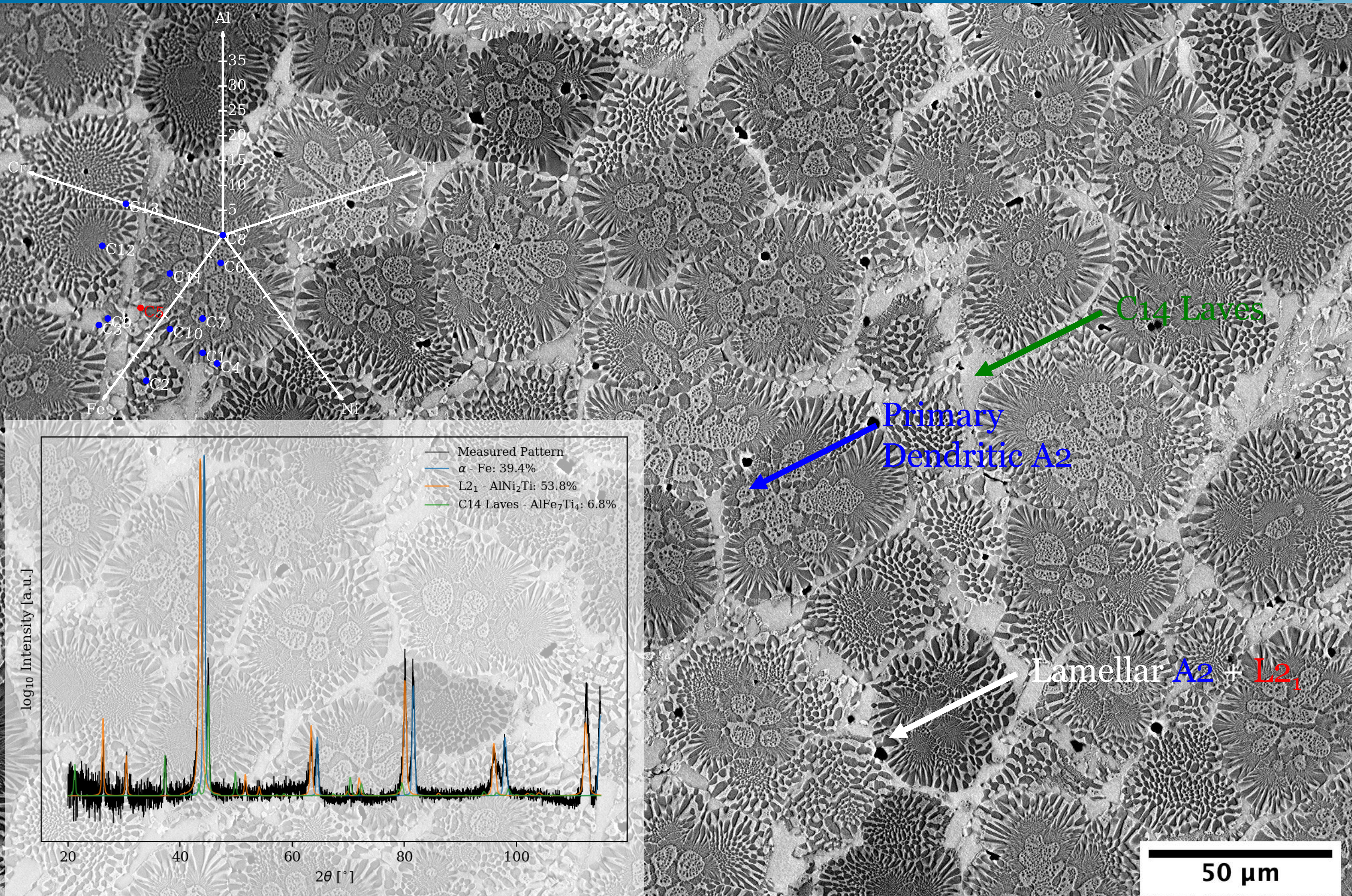
Solidus increases with Al content as $B2/L2_1$ is stabilized

What is happening to the Microstructure as Al content is increased?

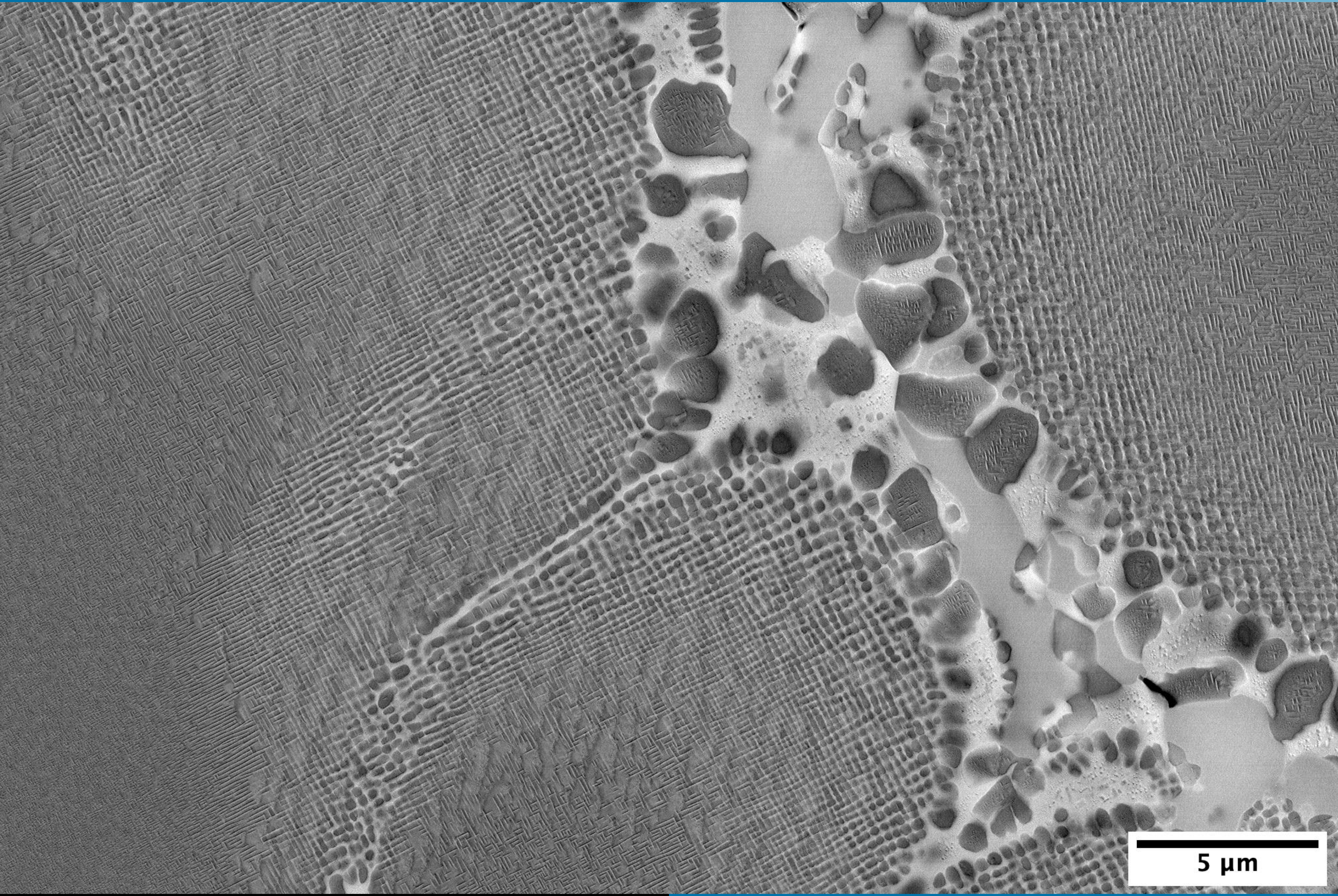
SEM Microstructures – C2 – $Al_{10}Cr_{20}Fe_{35}Ni_{25}Ti_{10}$



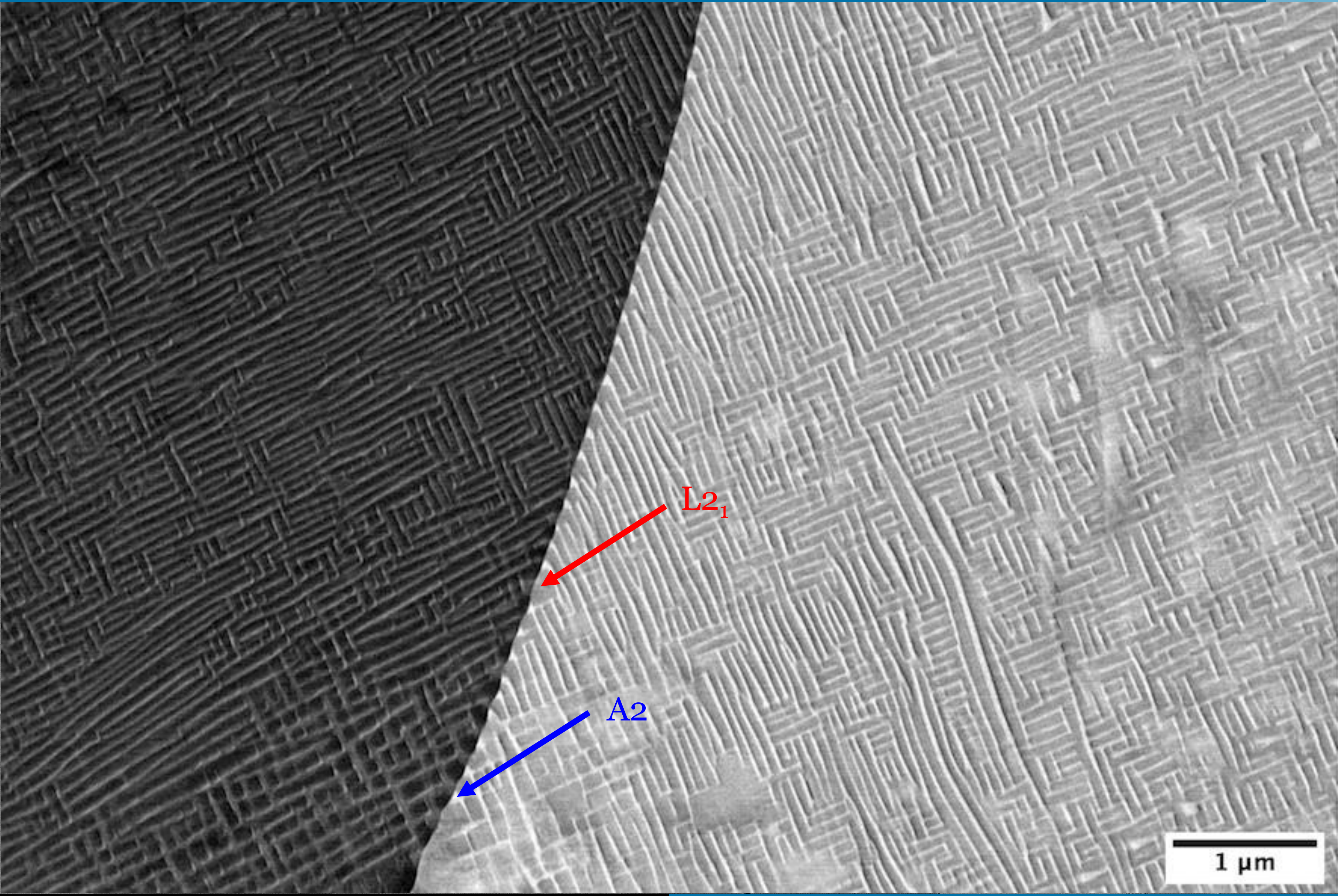
SEM Microstructures – C5 – $Al_{15}Cr_{20}Fe_{35}Ni_{15}Ti_{15}$



SEM Microstructures – C7 – $Al_{20}Cr_{10}Fe_{35}Ni_{20}Ti_{15}$



SEM Microstructures – C11 – $Al_{25}Cr_{15}Fe_{30}Ni_{20}Ti_{10}$



Following morphology trends observed:

I. **Bulk A2 regions with B2/L2$_1$ ordered phase precipitates** when the combined (Cr, Fe) content is higher than either the combined (Al, Ni, Ti) or (Fe, Ti) contents, and the Al content is below ~20 at.%

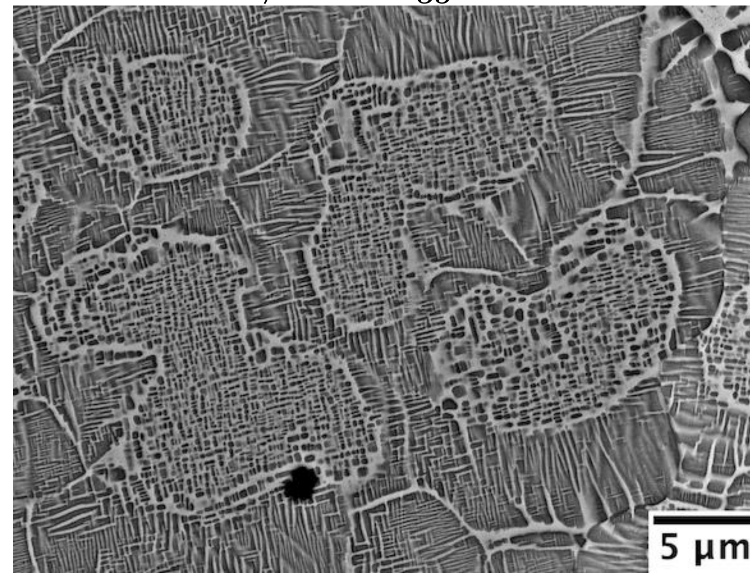
II. **Lamellar regions with alternating A2 and B2/L2$_1$ phase domains**—when the combined (Al, Ni, Ti) content is higher than either the combined (Fe, Ti) or (Cr, Fe) contents, or if the Al content is above ~20 at.%

III. C14 Laves regions when the combined (Fe, Ti) c
(

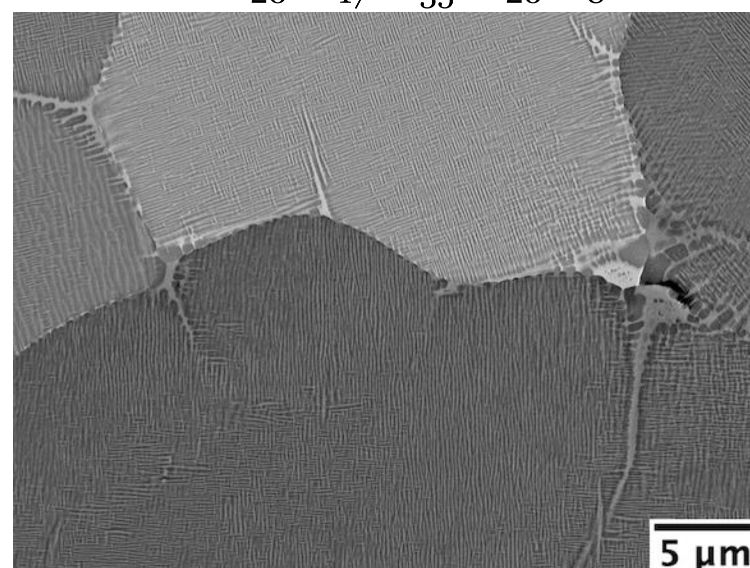
IV. I
combined (Fe, Ni) content is higher than (Fe, Ti), (Al, Ni, Ti), or (Cr, Fe).

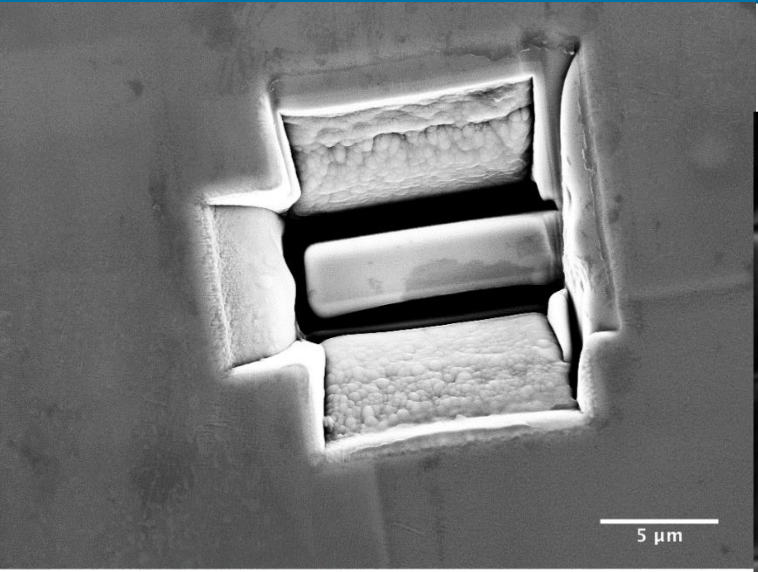
As Al content is increased, past 20 at.% and up to 30 at.%, the A2 and B2/L2$_1$ domains develop cuboidal faceted morphology due to coherent interfaces.

$Al_{17}Cr_{20}Fe_{35}Ni_{20}Ti_8$

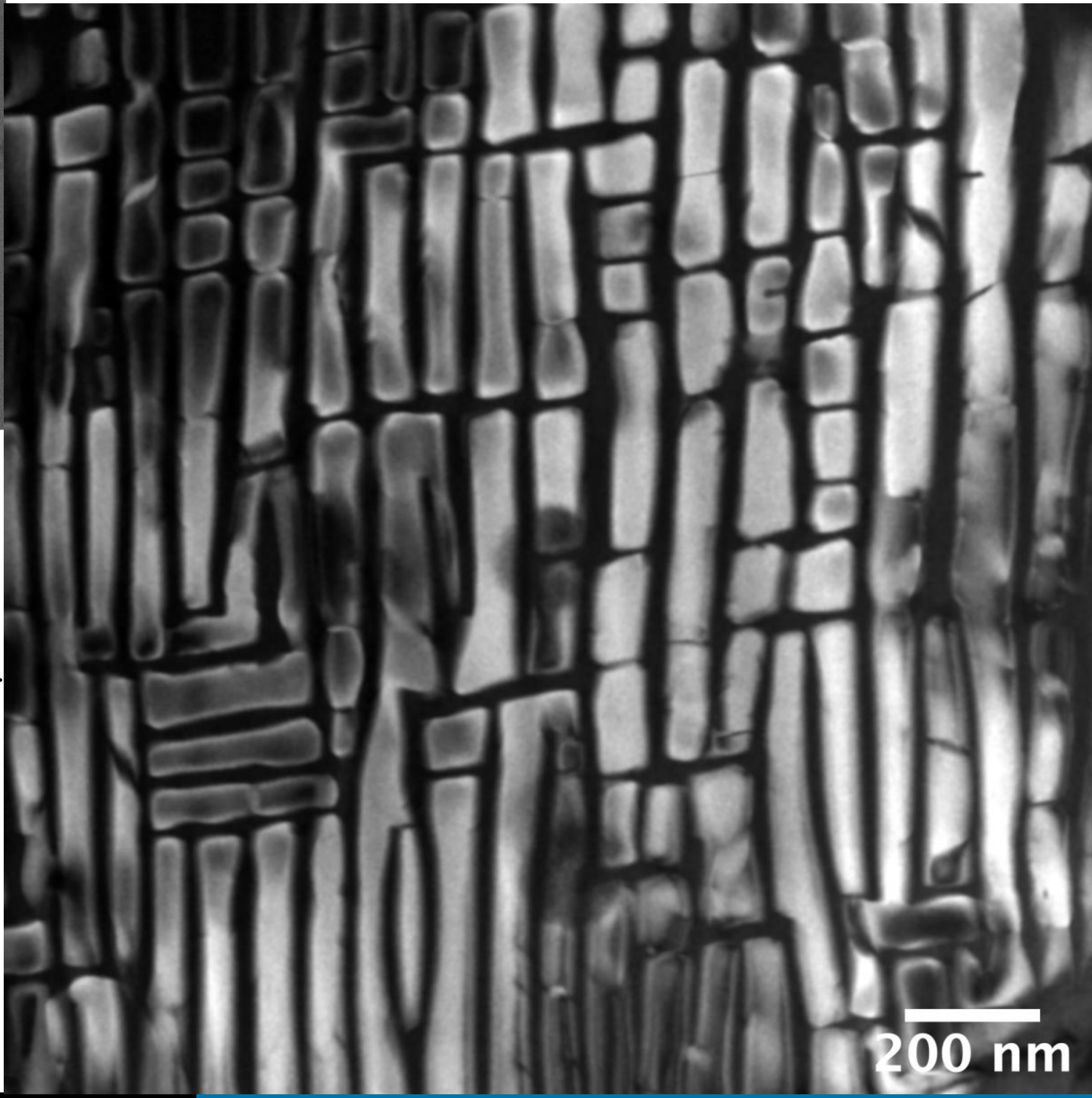


$Al_{20}Cr_{17}Fe_{35}Ni_{20}Ti_8$



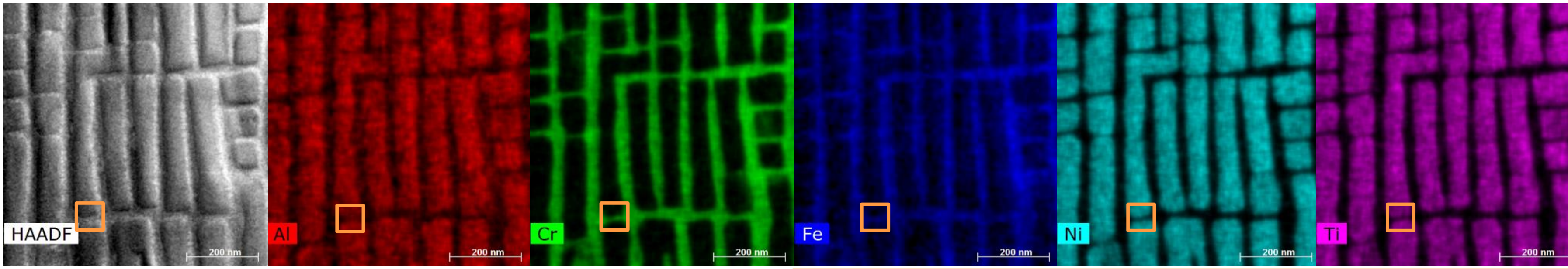


Side-view TEM lamella prepared by FIB



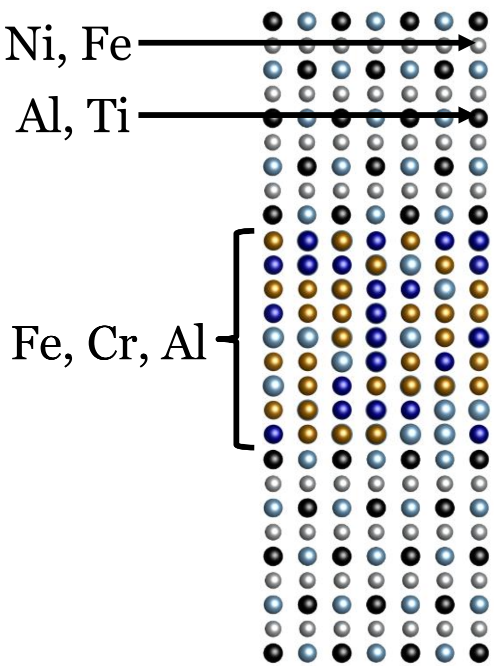
Two beam diffraction condition with <110> zone axis and $g_{111\text{-}L21}$ diffraction spot highlights $L2_1$ precipitates

STEM Analysis of C11

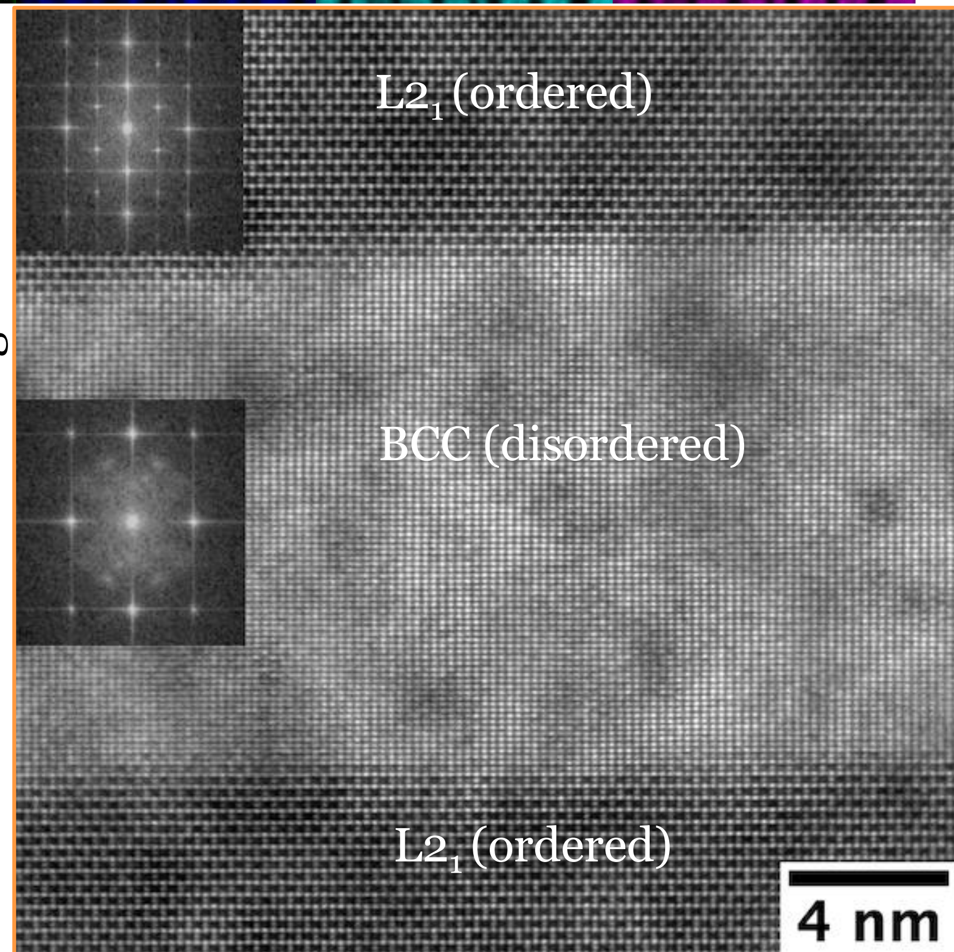


EDS-PCA gives quantified composition in separate phases

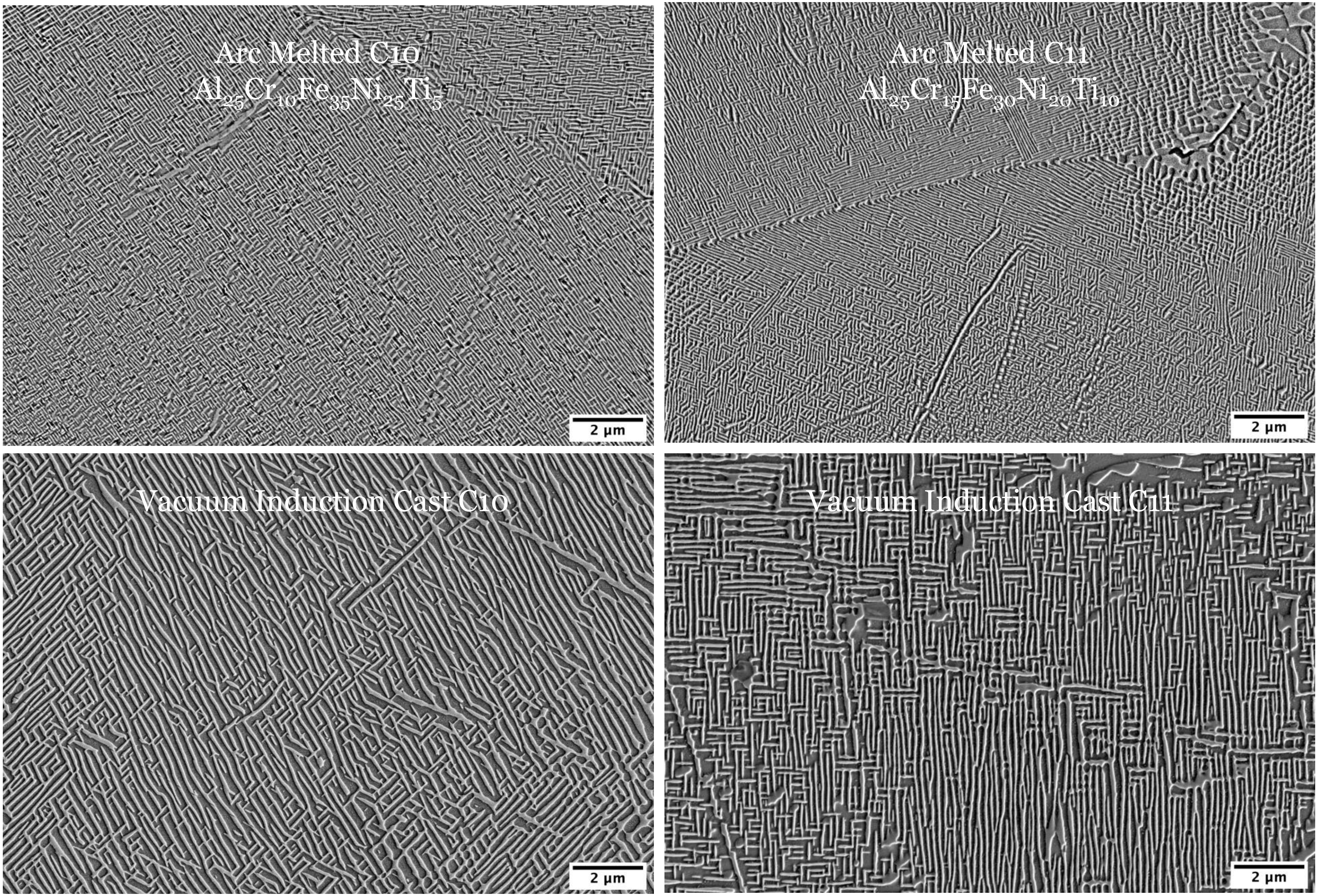
Component	Measured Composition				
	Al	Cr	Fe	Ni	Ti
A2	7	45	48	0	0
$L2_1$	25	0	25	32	18



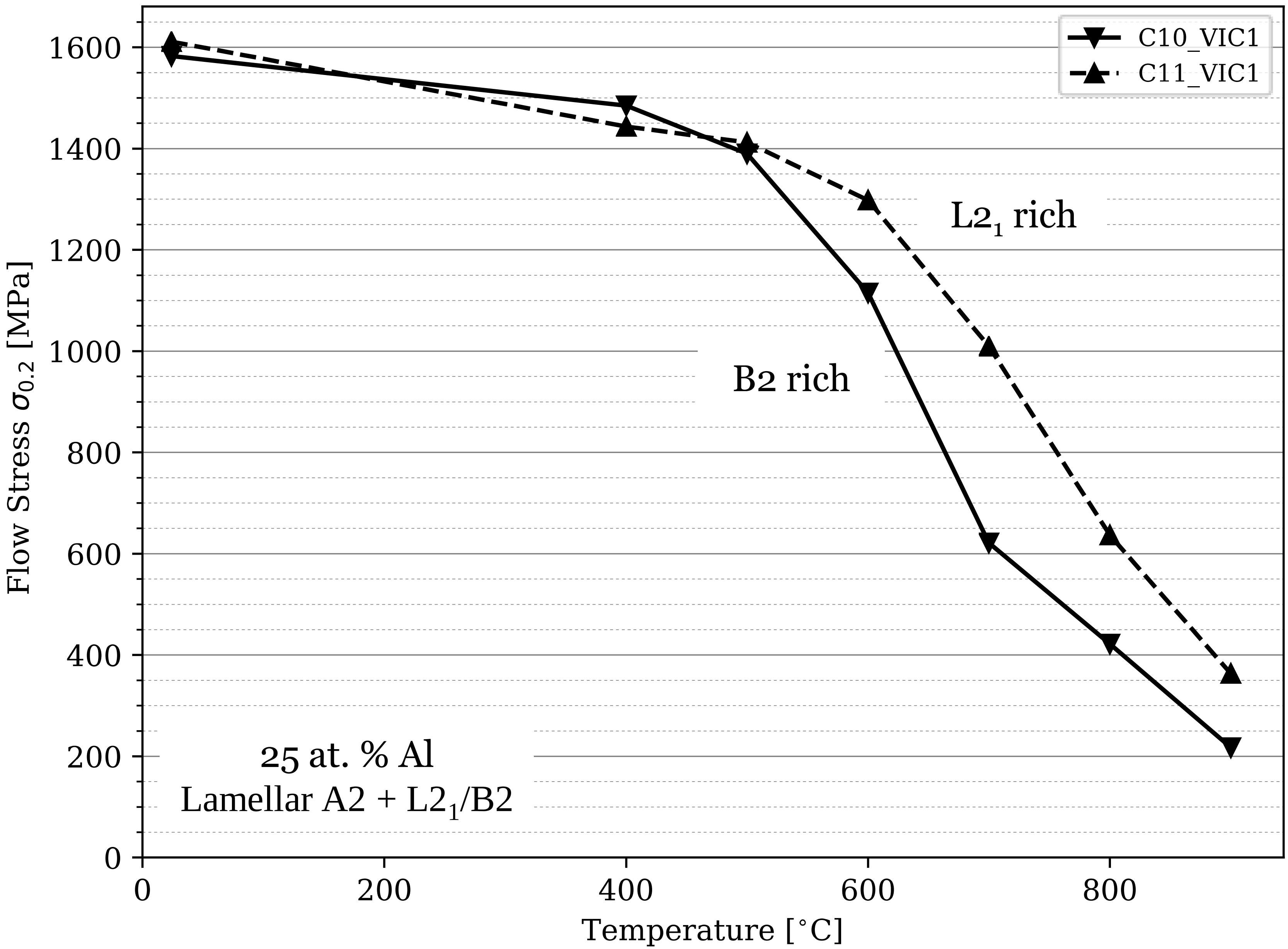
<110> zone axis
HAADF Image



Synthesis Technique Comparison – Microstructures



VIC Samples – High Temperature Compression Tests



Summary of Results

- **Successfully synthesized compositionally graded thin film**, allowing phase formation comparison with bulk alloys.

- Thin film trace with 35 at.% Fe and 20 at.%Cr showed **primarily disordered A2 formation for Al content from 15 to 25 at.%** and showed additional formation of nano-scale **ordered phase regions for Al contents between 20 and 25 at.%**.

- We observe the following primary phase formation in cast alloys with 30-35 at.% Fe:

  I. Bulk A2 regions with B2/$L2_1$ ordered phase precipitates when combined (Cr, Fe) content is higher than either (Al, Ni, Ti) or (Fe, Ti) contents, and Al content is below ~25 at.%.

  II. Lamellar regions with alternating A2 and B2/$L2_1$ domains when the (Al, Ni, Ti) content is higher than either (Fe, Ti) or (Cr, Fe), or if Al content is ~20-30 at.%.

  III. C14 Laves regions when the combined (Fe, Ti) content is higher than either (Cr, Fe) or (Al, Ni, Ti).

  IV. $L1_2$ regions when the (Fe, Ni) content is higher than either (Fe, Ti), (Al, Ni, Ti), or (Cr, Fe).

- Initial high temperature compression tests on as-cast alloys show **promising strength retention up to 900 C**.

Outlook – Next Steps

Future experiments will explore:

- Effects of various heat treatments on microstructure formation.
- Duplex FCC/BCC morphologies at higher Ni contents.
- Mechanical properties of a wider range of morphologies.
- Dependence of high temperature strength and creep resistance on morphology, through composition variations.
- Oxidation resistance of most promising compositions.

Thanks for your attention!

Special thanks to:

- Christian Liebscher (Guidance and discussion)
- Benjamin Breitbach (XRD)
- Frank Stein and Martin Palm (DSC and discussions)
- Dennis Klapproth (Sample synthesis)
- Leon Christiansen (Sample preparation)
- Thorsten Meiners (TEM)

Questions, Comments??

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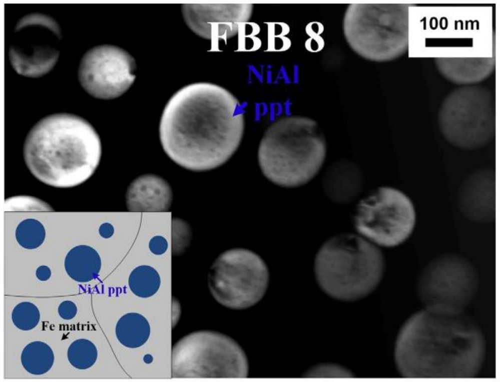
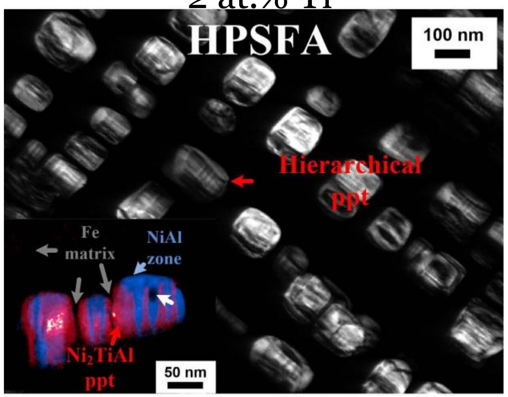
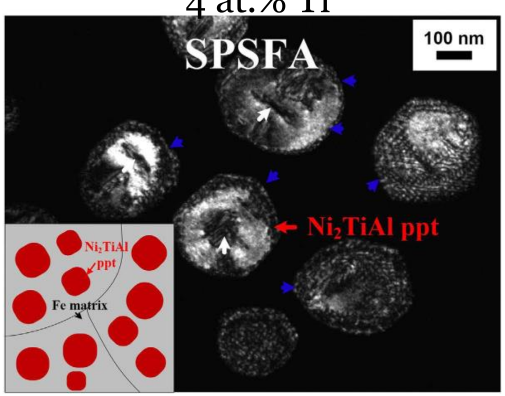
0 at.% Ti



2 at.% Ti



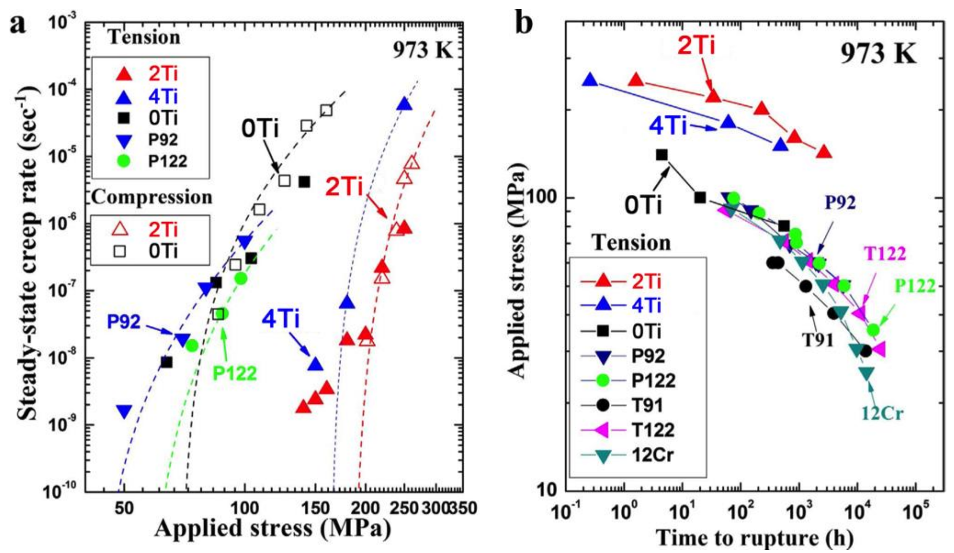
4 at.% Ti



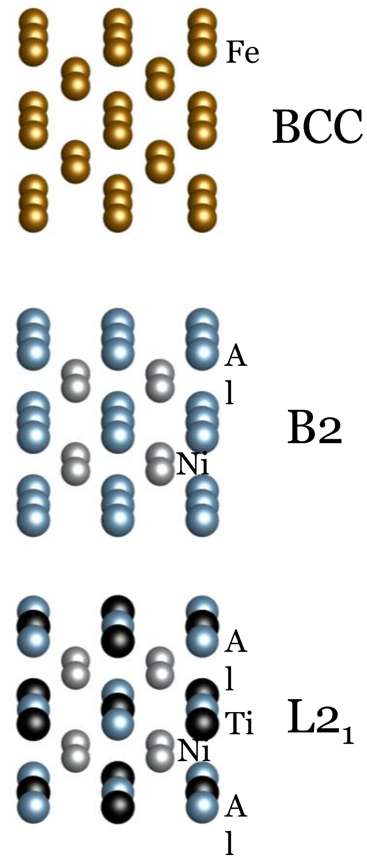
Inspiration: Fe-base Superalloys

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



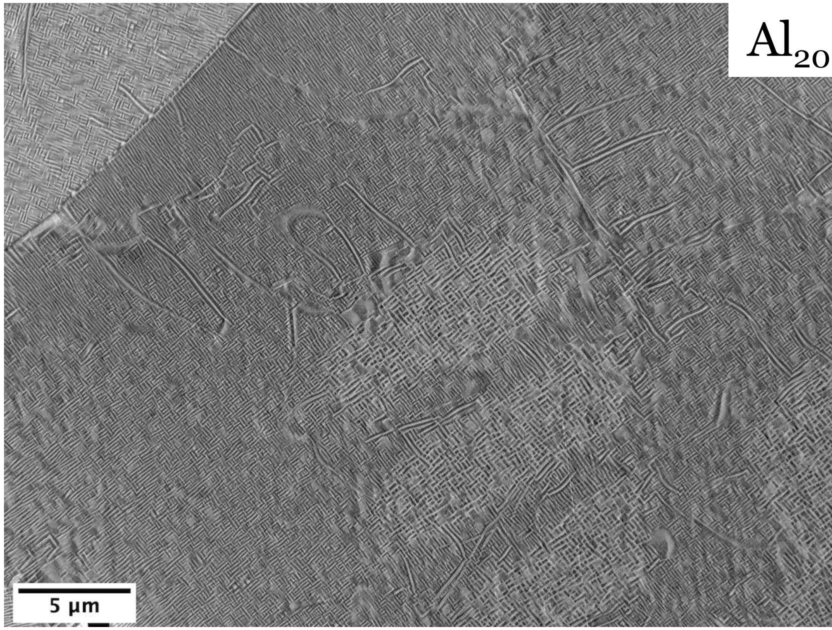
Song, Gian, et al. "Ferritic alloys with extreme creep resistance via coherent hierarchical precipitates." *Scientific reports* 5 (2015).

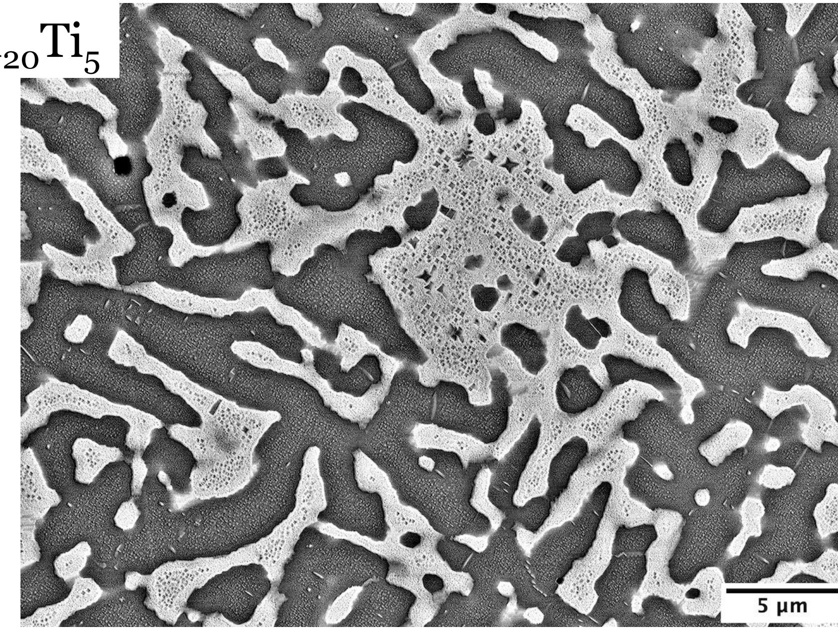


How far can creep resistance be improved by increasing B2/$L2_1$ phase fractions?

Homogenization – 1250 C, 2 hr – Water Quench

$Al_{20}Cr_{20}Fe_{35}Ni_{20}Ti_{5}$





$Al_{25}Cr_{15}Fe_{30}Ni_{20}Ti_{10}$