Early stage development of precipitation strengthened CCAs in the AlCrFeNiTi system for high temperature structural applications

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MAX-PLANCK-INSTITUT FÜR EISENFORSCHUNG

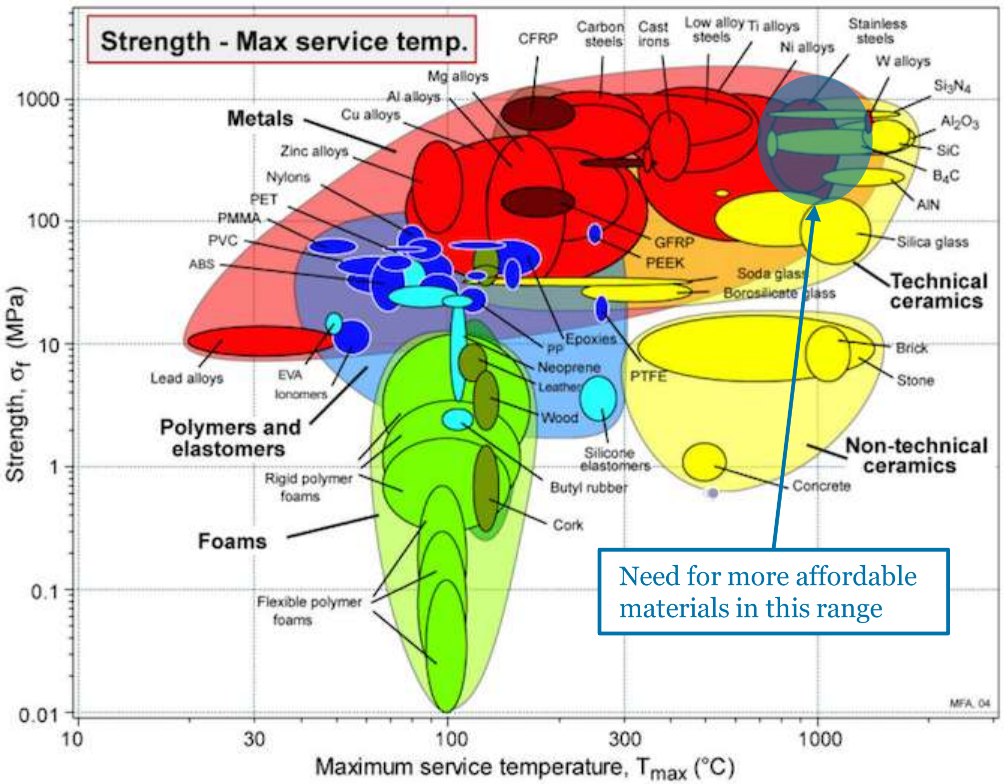
Silas Wolff-Goodrich
and Christian Liebscher

In Collaboration With:
Dr. Konda G. Pradeep
and Marshal Amalraj

MATERIALS CHEMISTRY | RWTH AACHEN UNIVERSITY

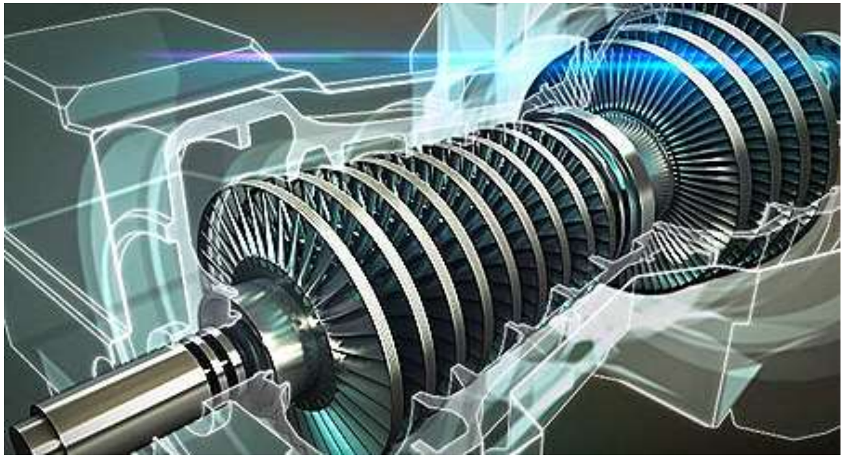
Outline

Novel high temperature materials needed!



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

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



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

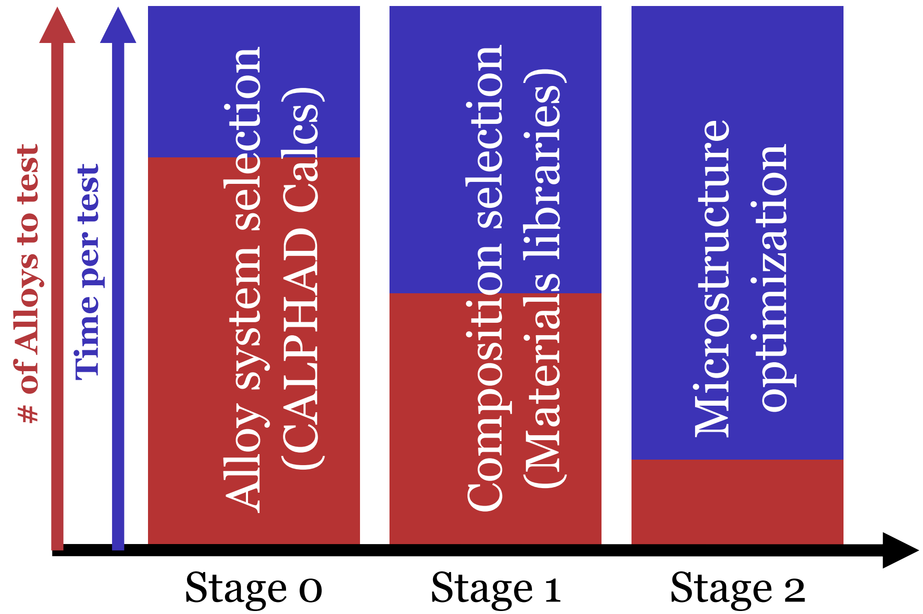
Alloys need:

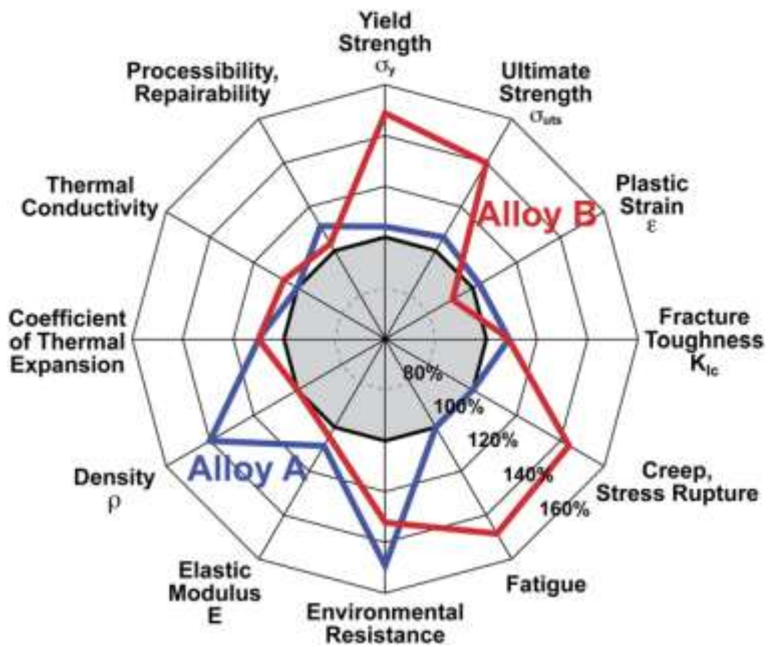
- Low-cost & low-density: **6-7 g/cm³**
- Creep resistance up to **900°C**
- Maximized solid solution strengthening & precipitation hardening

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

Compositionally Complex Alloy Development Methodology

"No longer safe along corners and edges of ternary phase diagrams, the materials community is now thrust into an uncharted, hyper-dimensional territory that is difficult to conceive, difficult to visualize, and difficult to explore systematically." [2]



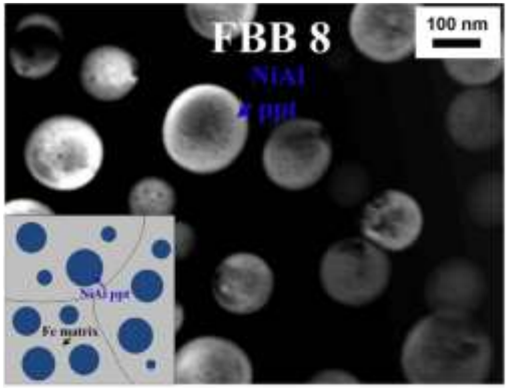


- Method most efficient for application specific search.
- "Spider plot" used for screening.
- Failure in one property = failure of alloy overall.

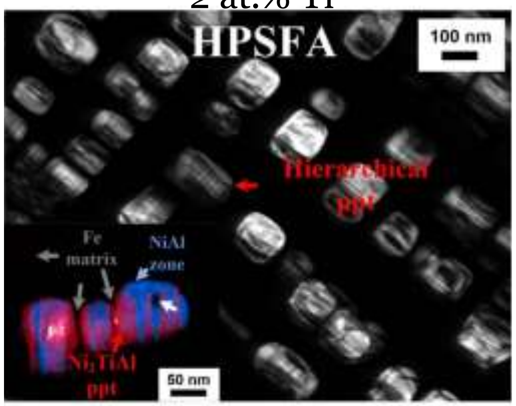
Miracle, Daniel, et al. "New strategies and tests to accelerate discovery and development of multi-principal element structural alloys." Scripta Materialia 127 (2017): 195-200.

Miracle, D. B., and O. N. Senkov. "A critical review of high entropy alloys and related concepts." *Acta Materialia* 122 (2017): 448-511.

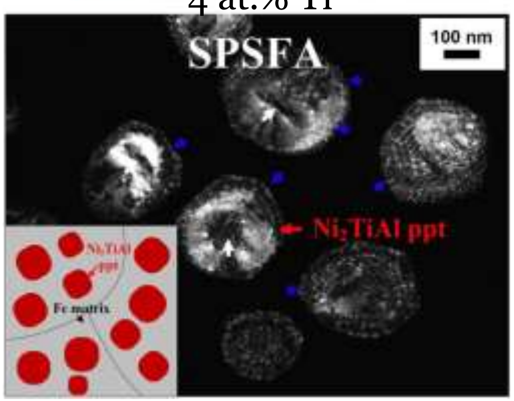
0 at.% Ti



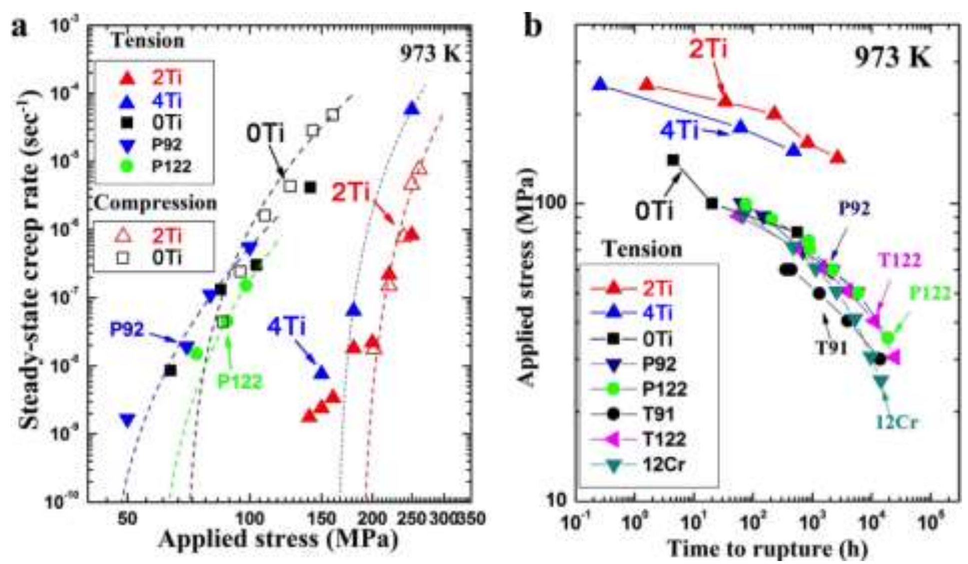
2 at.% Ti



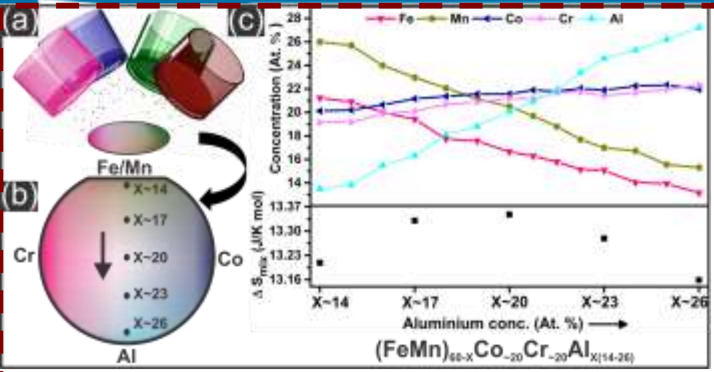
4 at.% Ti



- Coherent B2-NiAl precipitates in BCC matrix for AlCrFeNi
- Addition of Ti changes precipitate morphology and structure to combined $L2_1$-Ni_2TiAl and B2-NiAl
- Creep resistance of hierarchical B2/$L2_1$ structure greatly improved



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



Magnetron Sputtering ⟺ Arc Melting

Vacuum Induction Casting

Directional Solidification

High-throughput characterization:
- SEM/EDS/XRD
- Nano-hardness

In collaboration with Pradeep & Marshal at RWTH Aachen

Rapid Screening (MATERIALS CHEMISTRY | RWTH AACHEN UNIVERSITY)

Bulk Synthesis

DSC and Heat Treatment

Microstructural Characterization

Bulk Mechanical Testing

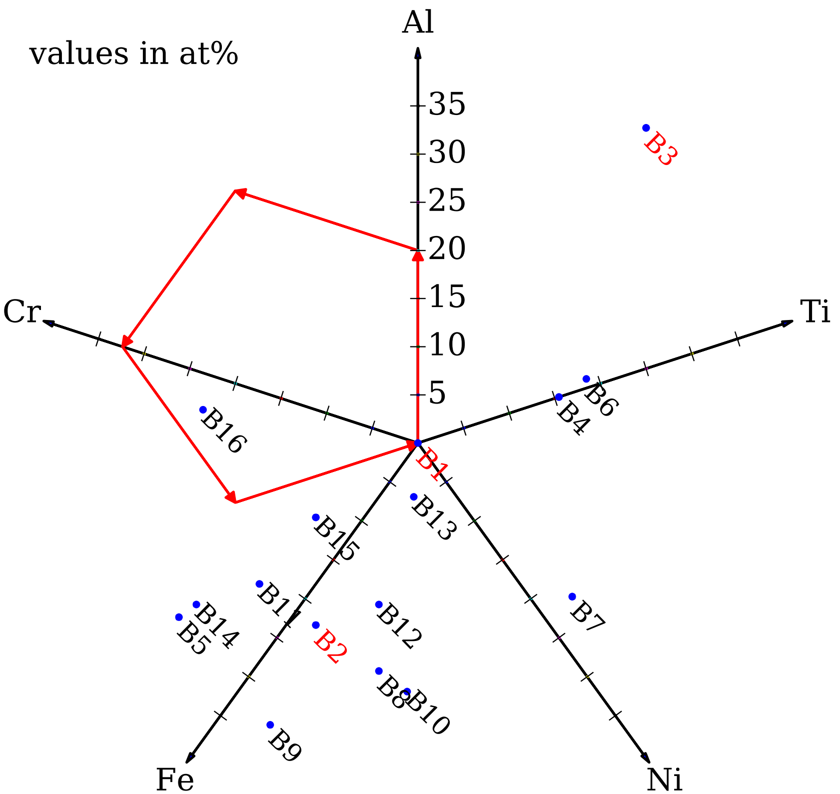
- XRD
- SEM
- (S)TEM
- 3D APT

- Compression
- Tension
- Nano- and micro-hardness
- Creep

Objectives
- BCC-CCAs with tailored **$B2/L2_1$-precipitates**
- Low-cost, low-density alloys: **6-7 g/cm³**
- Creep resistance up to **900°C**
- Maximized solid solution strengthening & precipitation hardening

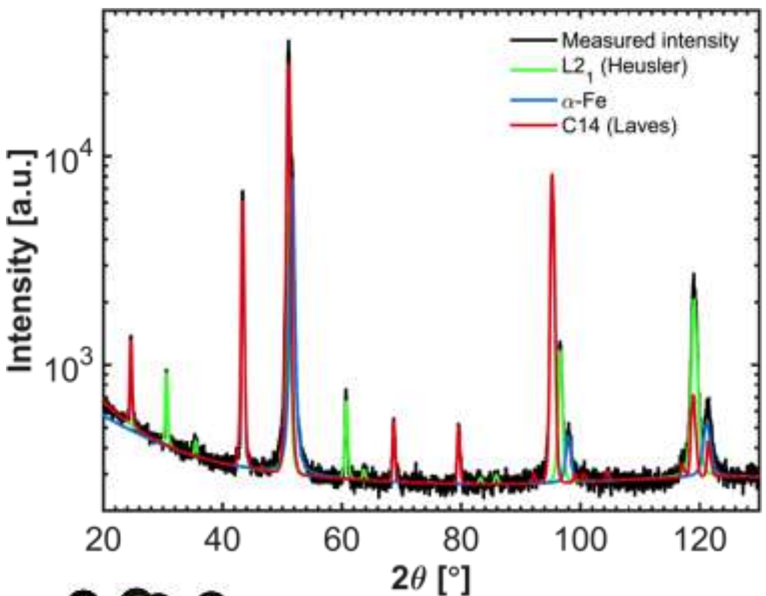
Sample ID	Composition (at%) Al	Cr	Fe	Ni	Ti	Solid Solution Rating (1-5)	Precipitates Rating (1-5)
B1	20	20	20	20	20	BCC 5	C_{14},L_{21},B_2 3
B2	25	10	35	25	5	BCC 5	L_{21} 3
B3	35	10	10	10	35	Hexagonal 1	None 1
B4	35	10	10	35	10	- -	- -
B5	10	30	30	20	10	- -	- -
B6	17.5	15	17.5	15	35	- -	- -
B7	5	15	25	20	35	- -	- -
B8	10	15	35	20	20	- -	- -
B9	10	20	35	25	10	- -	- -
B10	15	10	35	25	15	- -	- -
B11	15	20	35	15	15	- -	- -
B12	20	10	35	20	15	- -	- -
B13	20	10	35	10	25	- -	- -
B14	20	20	35	20	5	- -	- -
B15	25	15	30	20	10	- -	- -
B16	25	20	35	5	15	- -	- -

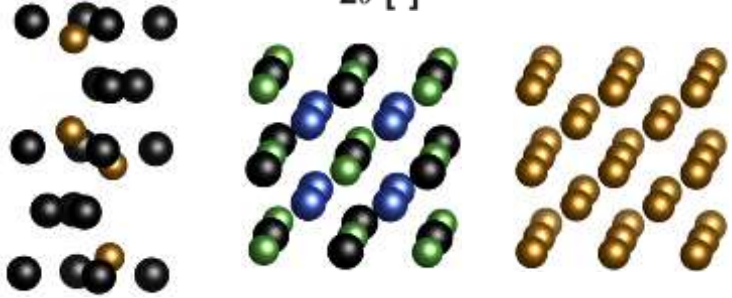
Planned Rating Categories:
- Solid solution structure
- Precipitation phases
- Density
- Castability
- Cost
- Hardness
- Ductility



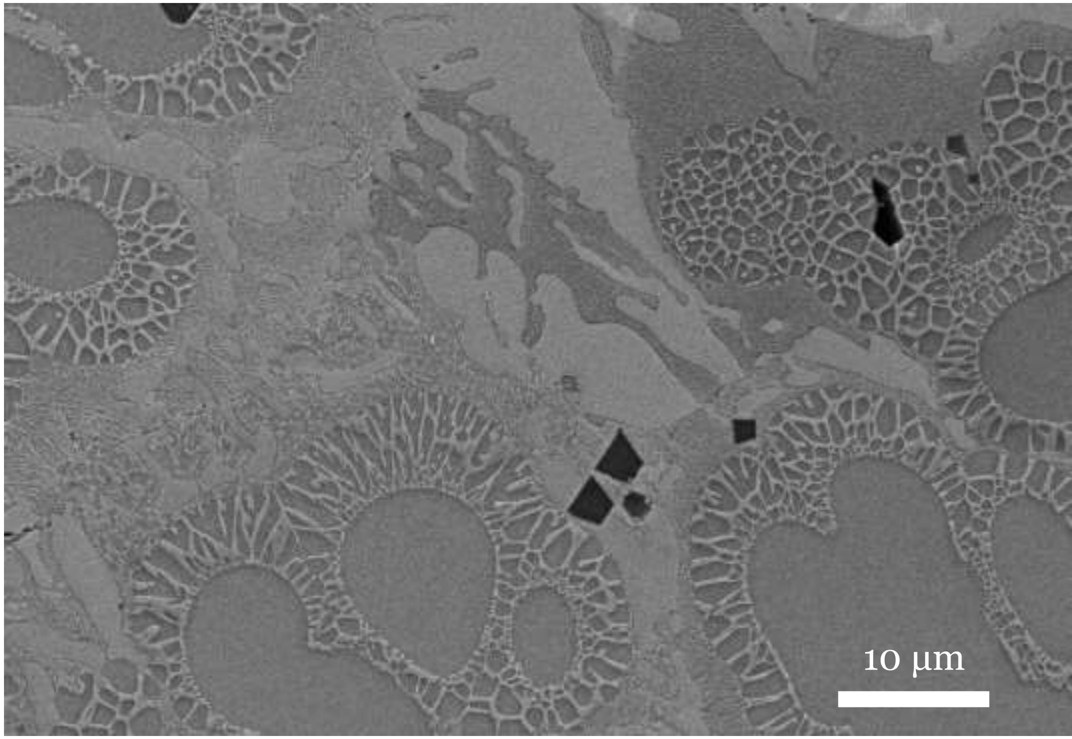
Early Results – B1 ($Al_{20}Cr_{20}Fe_{20}Ni_{20}Ti_{20}$)

- DSC results indicate that the first major thermal effect occurs around 1150 °C. Homogenization will be conducted at 1200 °C and annealing at 900 °C.
- As-cast material:





C14 $L2_1$ α-Fe



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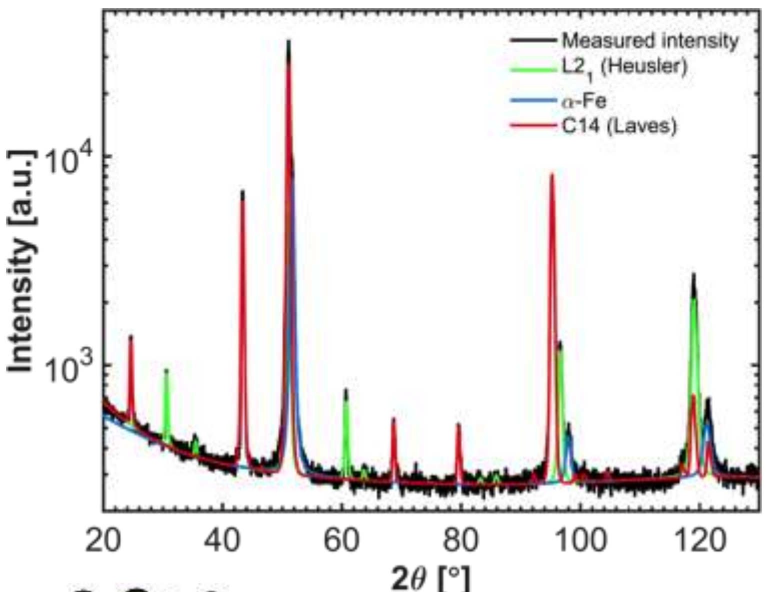

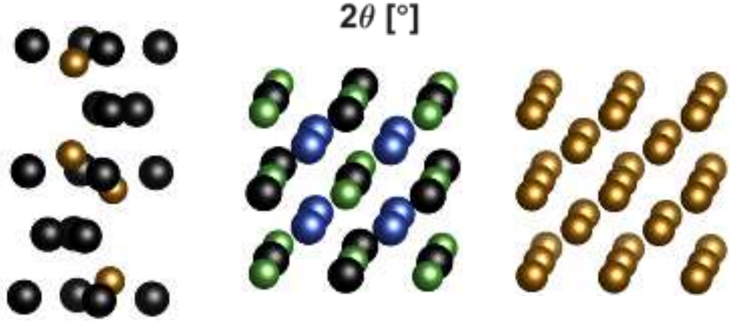

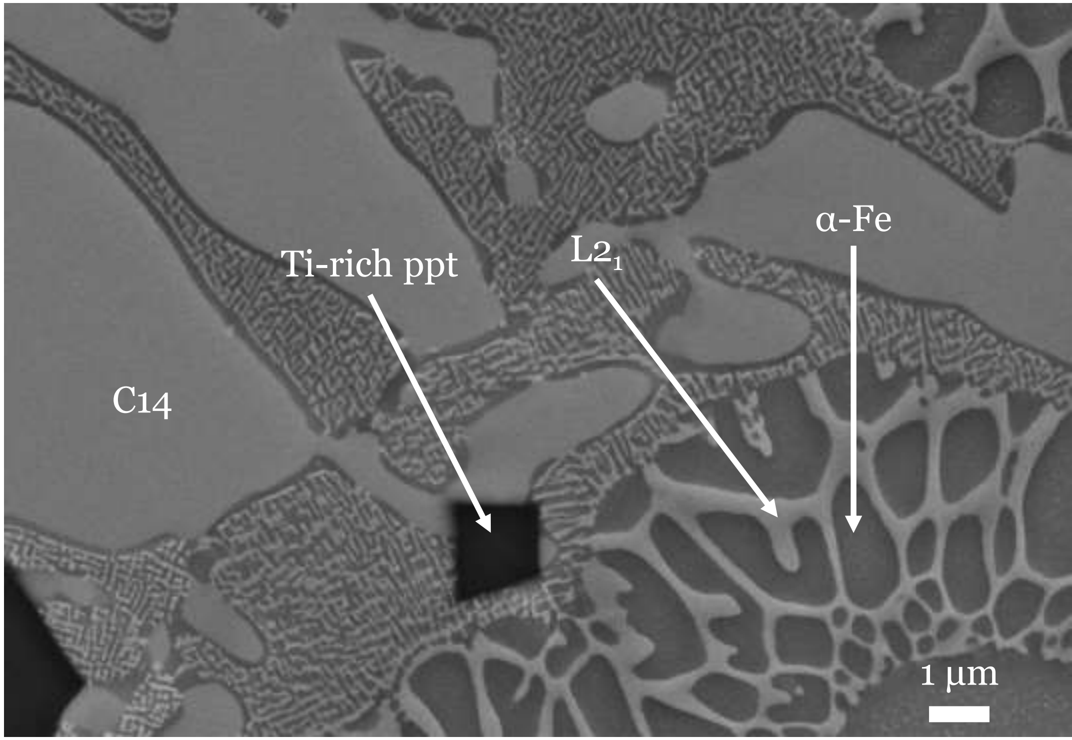

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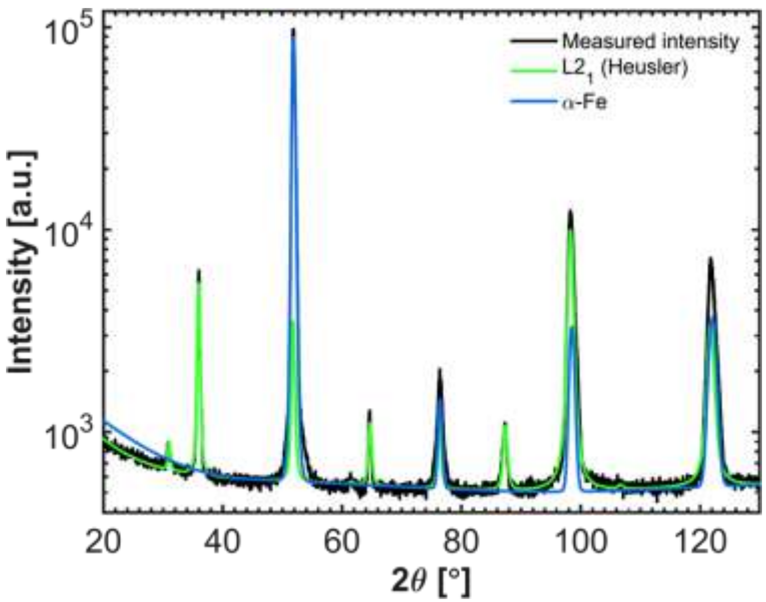
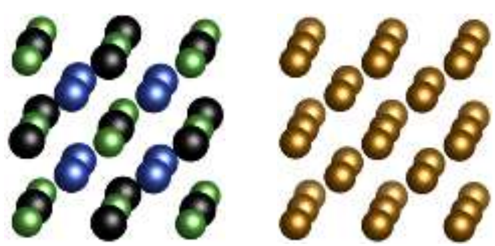
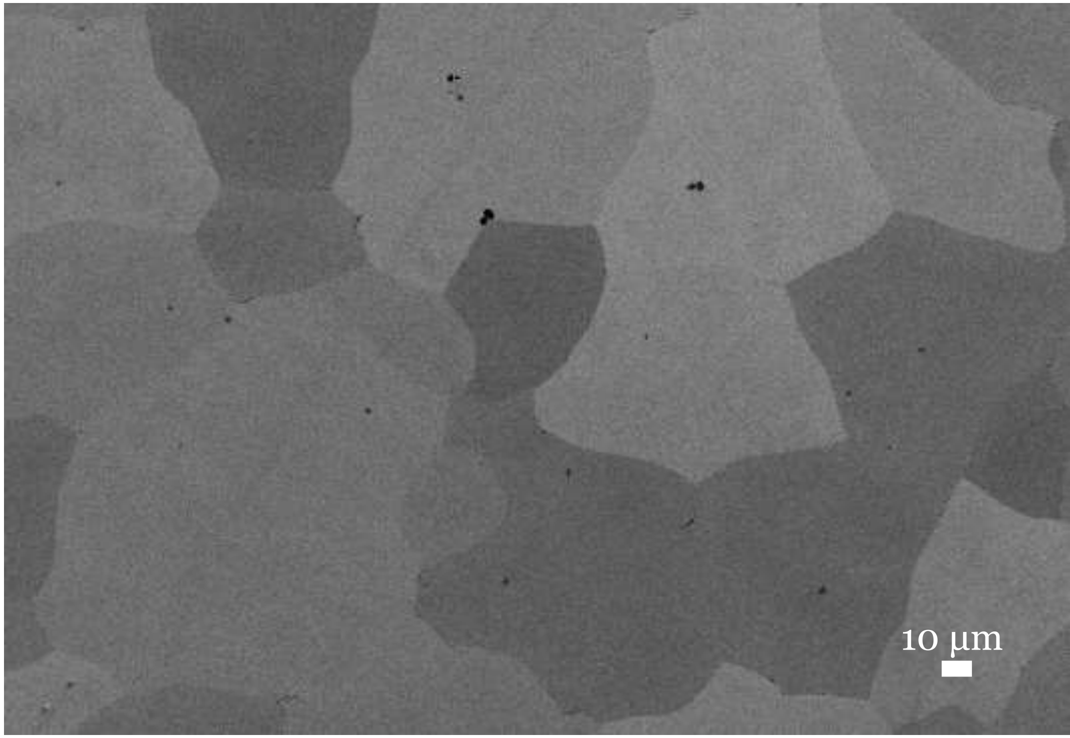




L2$_1$ α-Fe



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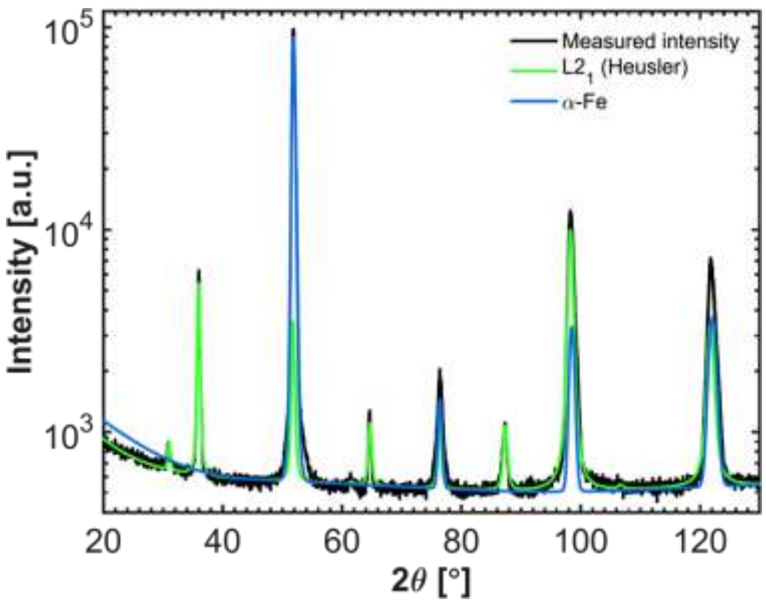
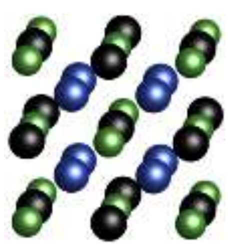
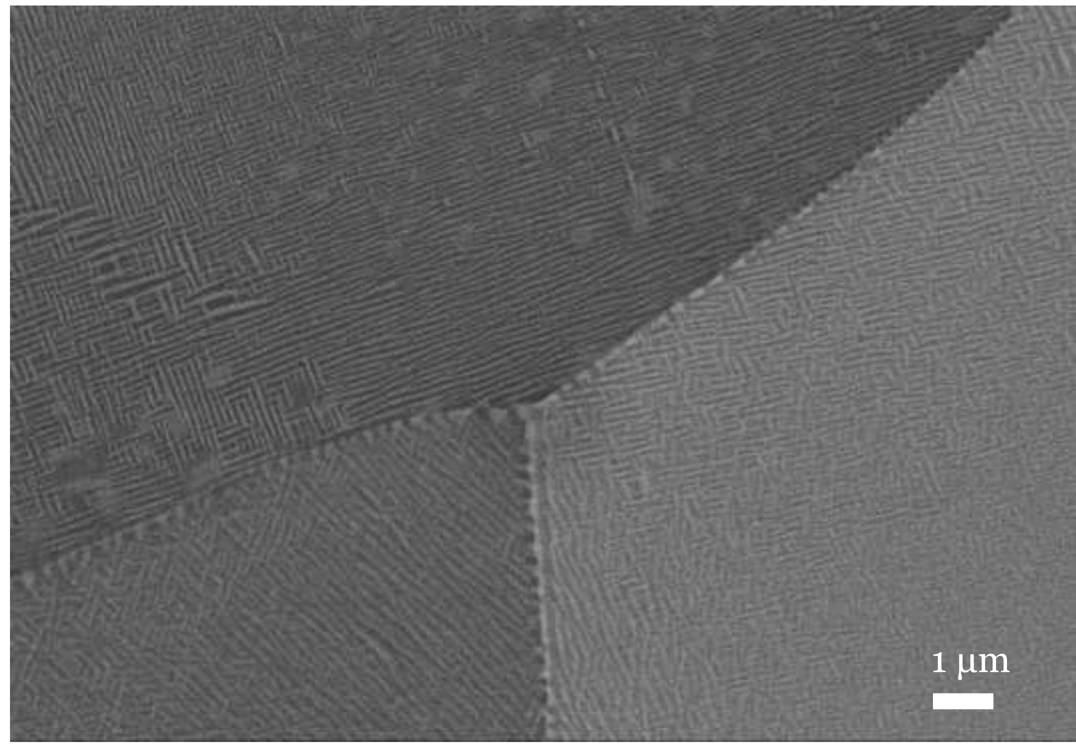




$L2_1$

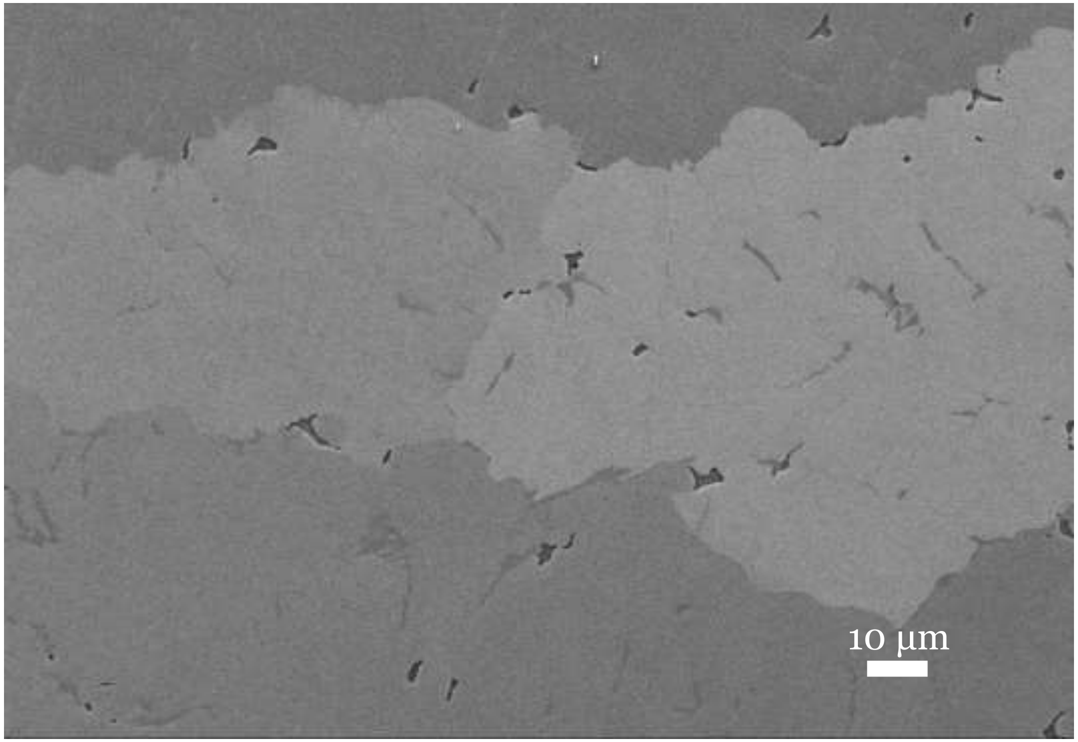
α-Fe



Early Results – B3 ($Al_{35}Cr_{10}Fe_{10}Ni_{10}Ti_{35}$)

- As-cast material:

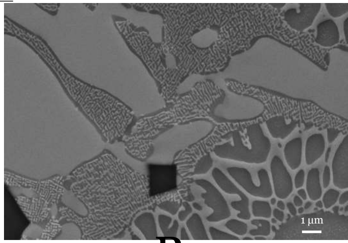
Single Hexagonal Phase detected



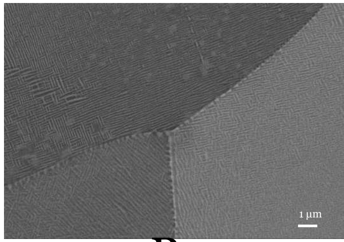
Results Summary

Sample ID	Composition (at%) Al	Cr	Fe	Ni	Ti	Solid Solution Rating (1-5)	Precipitates Rating (1-5)	Density (g/cm^3) Rating (1-5)	Castibility Rating (1-5)	Cost ($/kg) Rating (1-5)	Hardness (MPa) Rating (1-5)	Ductility (% el.) Rating (1-5)	Average (1-5)
B1	20	20	20	20	20	BCC 5	C_{14},L_{21},B_2 3	5.81 3	Whole 5	- -	- -	- -	-
B2	25	10	35	25	5	BCC 5	L_{21} 3	6.17 2	Whole 5	- -	- -	- -	-
B3	35	10	10	10	35	Hexagonal 1	None 1	4.60 5	Fractured 1	- -	- -	- -	-
B4	35	10	10	35	10	- -	- -	5.49 3	Fractured 1	- -	- -	- -	-
B5	10	30	30	20	10	- -	- -	6.73 1	Whole 5	- -	- -	- -	-
B6	17.5	15	17.5	15	35	- -	- -	5.45 3	Fractured 1	- -	- -	- -	-
B7	5	15	25	20	35	- -	- -	6.15 2	Fractured 1	- -	- -	- -	-
B8	10	15	35	20	20	- -	- -	6.42 2	Whole 5	- -	- -	- -	-
B9	10	20	35	25	10	- -	- -	6.83 1	Whole 5	- -	- -	- -	-
B10	15	10	35	25	15	- -	- -	6.38 2	Whole 5	- -	- -	- -	-
B11	15	20	35	15	15	- -	- -	6.25 2	Whole 5	- -	- -	- -	-
B12	20	10	35	20	15	- -	- -	6.05 2	Whole 5	- -	- -	- -	-
B13	20	10	35	10	25	- -	- -	5.64 3	Partial 3	- -	- -	- -	-
B14	20	20	35	20	5	- -	- -	6.37 2	Whole 5	- -	- -	- -	-
B15	25	15	30	20	10	- -	- -	5.92 3	Whole 5	- -	- -	- -	-
B16	25	20	35	5	15	- -	- -	5.61 3	Whole 5	- -	- -	- -	-

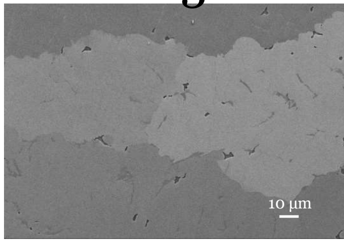
B1



B2



B3



Silas Wolff-Goodrich s.wolffgoodrich@mpie.de

- Priorities for the next steps in the project include:
  - Obtain thin film samples from collaborators in Aachen
  - Standardize SEM and XRD measurement and analysis procedures
  - Refine alloy rating scheme
  - Characterize heat treated samples and refine heat treatment
  - Begin mechanical property screening

Thanks for your attention!

Questions??

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Sources

[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.

[2] Miracle, D. B., and O. N. Senkov. "A critical review of high entropy alloys and related concepts." *Acta Materialia* 122 (2017): 448-511.

[3] Vo, Nhon Q., et al. "Creep properties and microstructure of a precipitation-strengthened ferritic Fe–Al–Ni–Cr alloy." *Acta Materialia* 71 (2014): 89-99.

[4] C.H. Liebscher, V.R. Radmilović, U. Dahmen, N.Q. Vo, D.C. Dunand, M. Asta, G. Ghosh, Acta Materialia, 92 (2015) 220-232.

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

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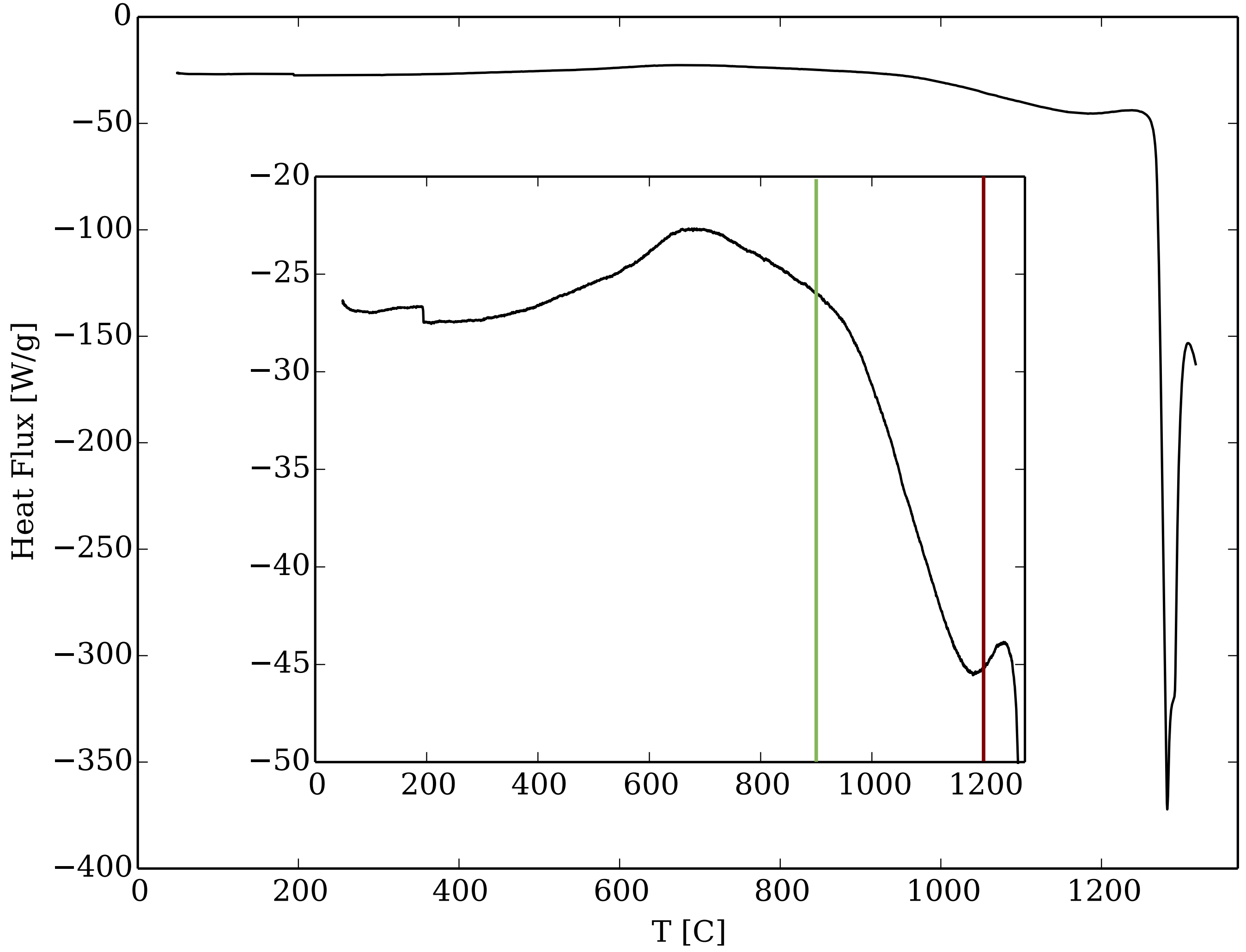
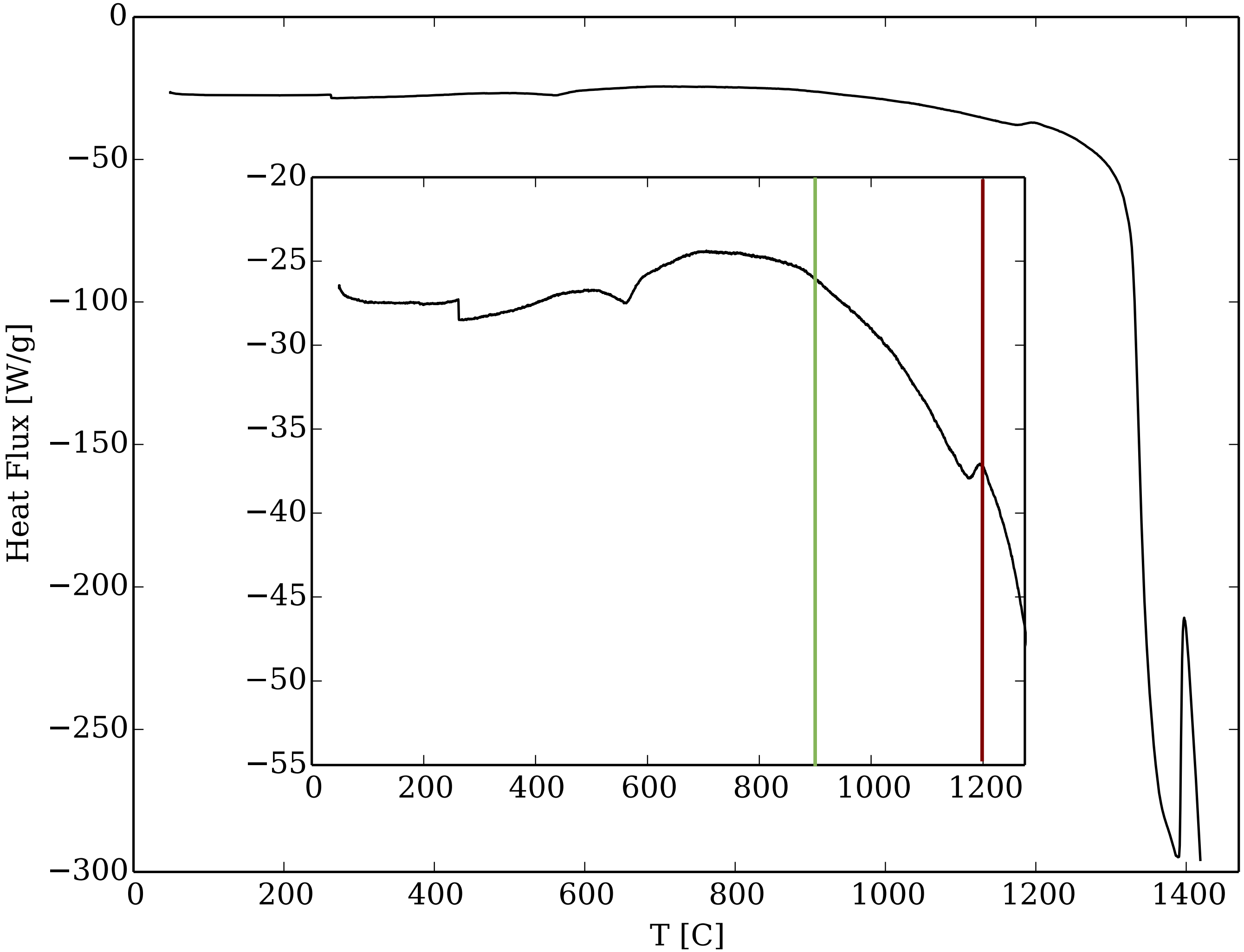


Early Results – B2 ($Al_{25}Cr_{10}Fe_{35}Ni_{25}Ti_5$)

- For Comparison with Equimolar alloy, homogenization will be conducted at 1200 °C and annealing at 900 °C.



HEA and CCA Definitions and Differences

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

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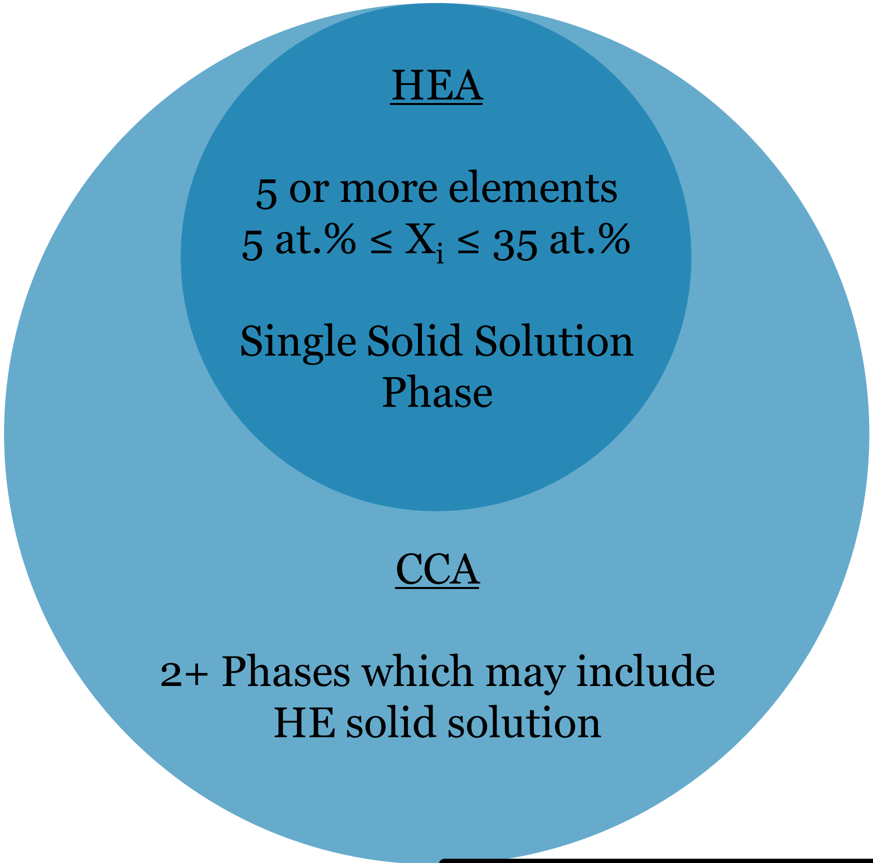
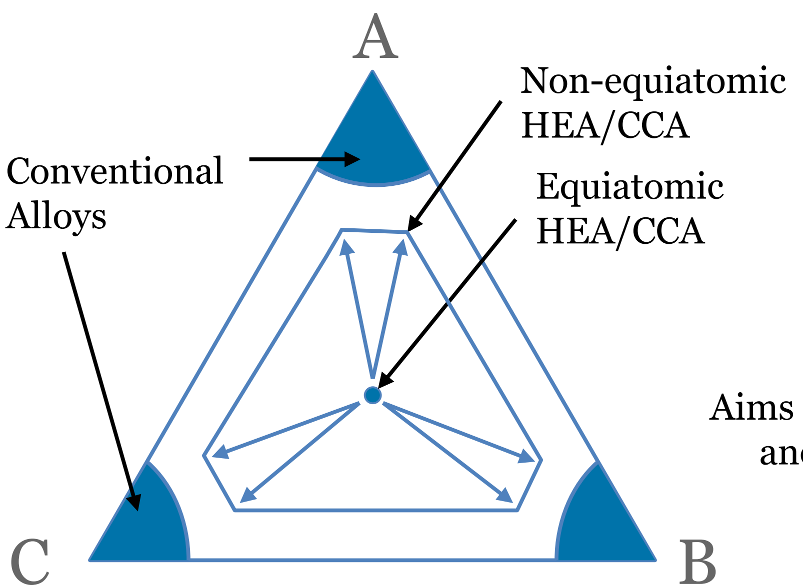




HEA Branch

DFG Deutsche Forschungsgemeinschaft

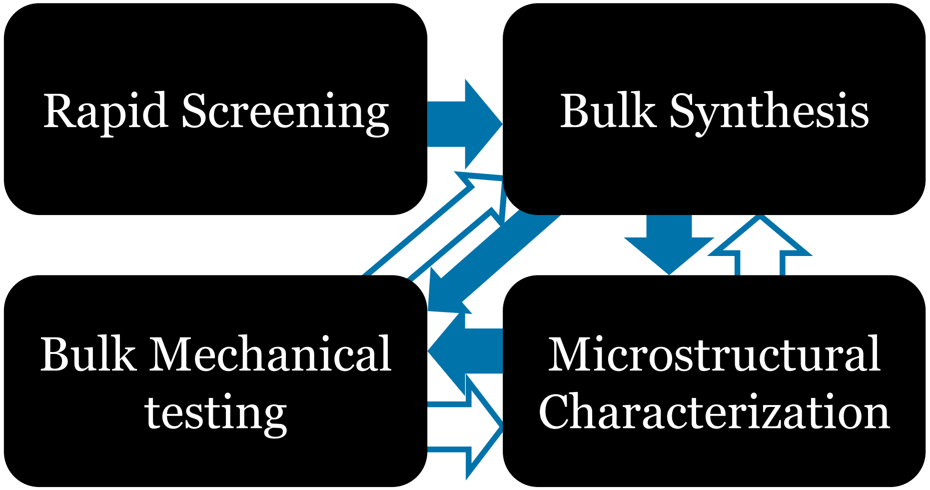
Aims at understanding structure and properties resulting from high entropy effect

CCA Branch

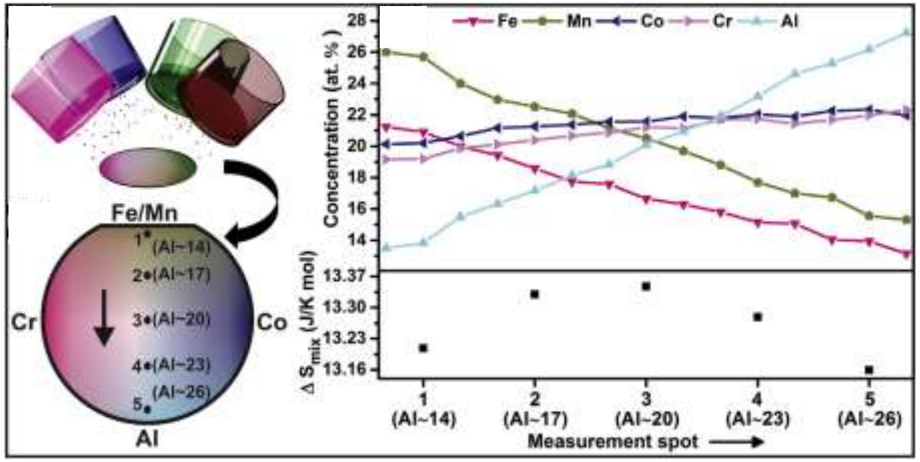
DFG Deutsche Forschungsgemeinschaft

Aims to identify and tailor chemistry and microstructural features for optimized mechanical properties in promising alloy systems

Work Flow



Thin Film Magnetron Sputtering



Composition selection

In collaboration with Pradeep & Marshal at RWTH Aachen

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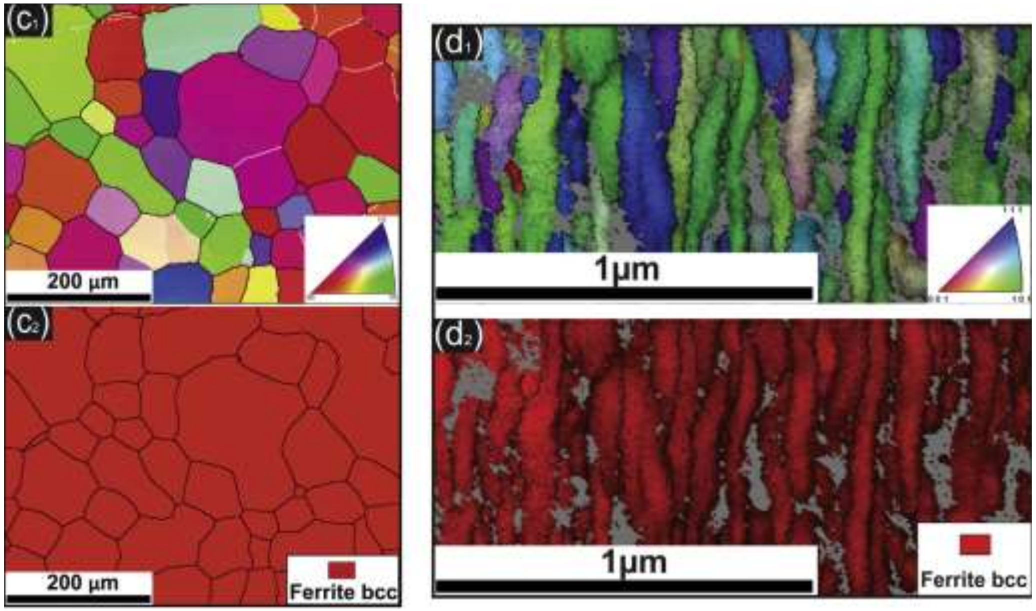


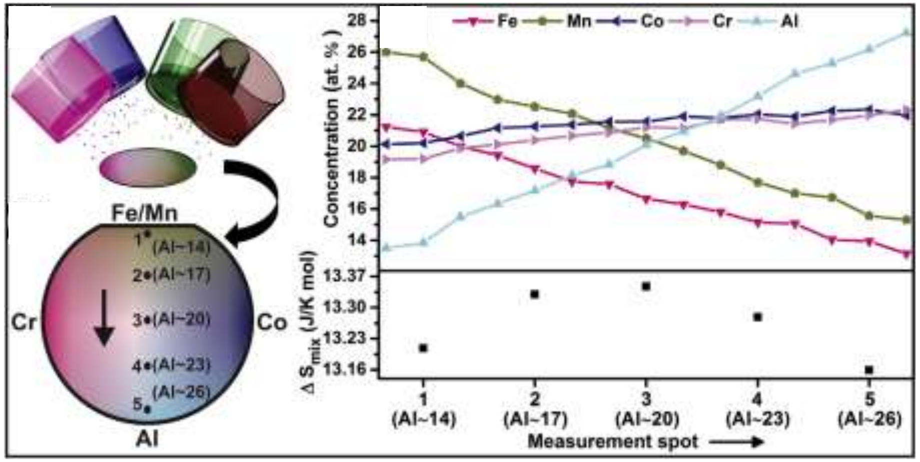
High-throughput characterization:

- SEM/EDS,EBSD
- XRD
- Nano-hardness

Marshal, A., et al. "Combinatorial synthesis of high entropy alloys: Introduction of a novel, single phase, body-centered-cubic FeMnCoCrAl solid solution." Journal of Alloys and Compounds 691 (2017): 683-689.

Rapid Screening

Thin Film Magnetron Sputtering



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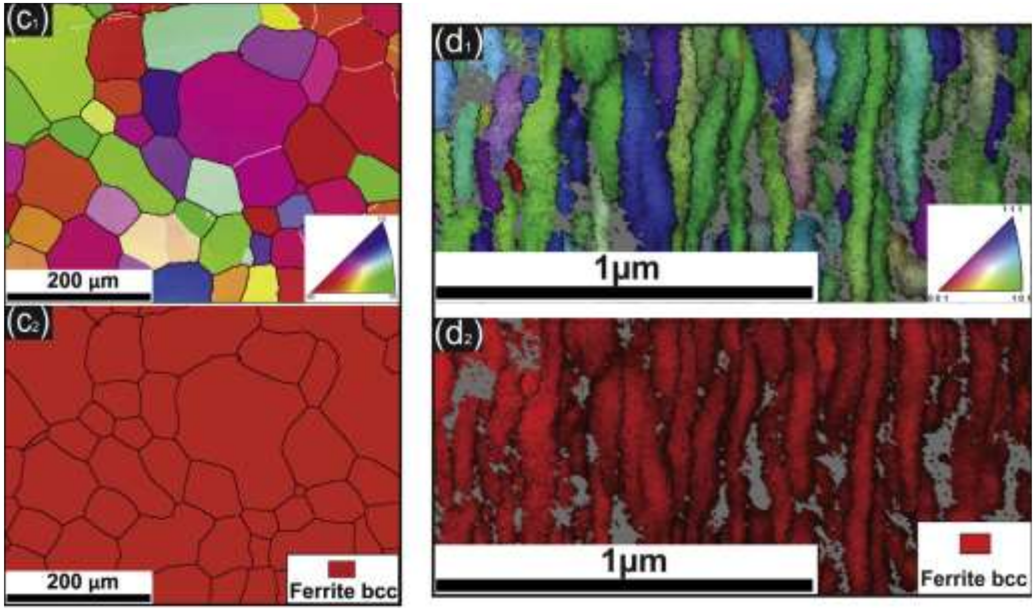

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Rapid Screening → Bulk Synthesis

Combinatorial Vacuum Induction Casting

Ma, Duancheng, et al. "Phase stability of non-equiatomic CoCrFeMnNi high entropy alloys." Acta Materialia 98 (2015): 288-296.

<u>Arc Melting</u>

- Use high throughput characterization to compare with thin films
- Far from equilibrium solidification

<u>Vacuum Induction Casting</u>

- Produces enough material for bulk mechanical testing
- Can produce several compositions in single cast

<u>Directional Solidification</u>

- For testing single crystal properties of most promising alloys

Bulk material to be combined with DSC results to select heat treatment schedules

Microstructural characterization will be used to direct alloy selection

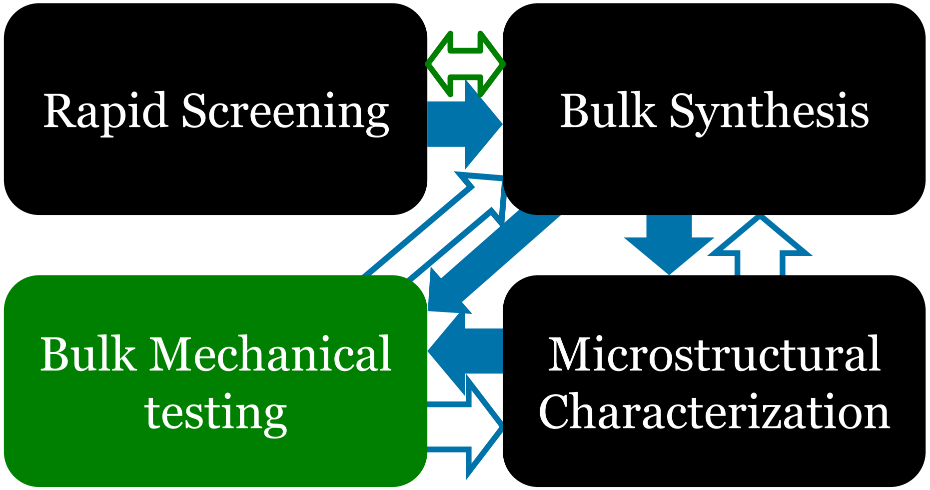
Work Flow

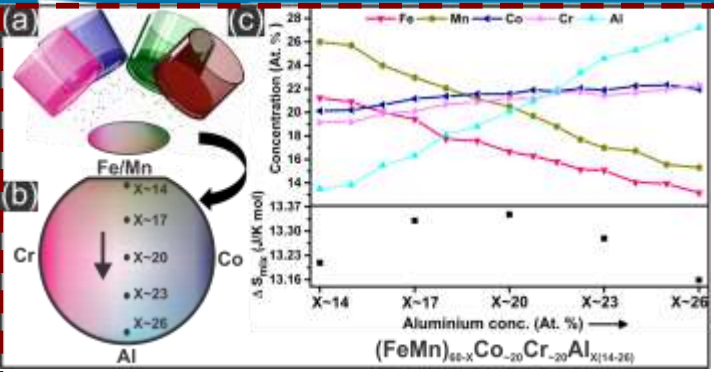
Rapid Screening → Bulk Synthesis

Microstructural Characterization

- XRD
- SEM/EDS
- (S)TEM
- 3D APT

Work Flow


Rapid Screening
Bulk Synthesis
Bulk Mechanical testing
Microstructural Characterization



Magnetron Sputtering ⇔ Arc Melting

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