Synthesis of particle strengthened CoCrFeNi-based HEA-CCA via laser-powder bed fusion process

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Outline

- PaCCman project

- Title: Particle-strengthened Compositionally Complex Alloys (PaCCman)
  - Interlinking powder synthesis, additive manufacturing, microstructure evolution and deformation mechanisms

- Objectives:
  (a) Synthesis of p-CCA via gas-atomization and L-PBF process
  (b) Investigation of the influence of L-PBF process on micro- and nanostructure
  (c) Study of deformation mechanisms of p-CCA synthesized by L-PBF process

- Team:

Powder Technology	+	Additive Manufacturing	+	Deformation Mechanism
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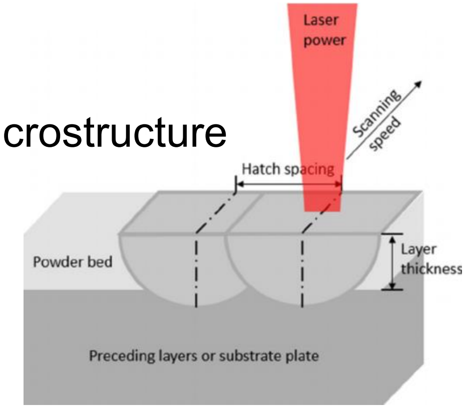
- Laser-powder bed fusion process

(1) Near net-shape manufacturing

(2) Rapid solidification

→ Reduction of segregation, extension of solid solubility, fine microstructure



- Research interests

Bulk synthesis of <u>particle strengthened CoCrFeNi-based CCA</u> via L-PBF process

(a) Nitrides in HEA/CCA matrix
(b) B2 in A1 matrix, or (c) B2 in A2 matrix

CoCrFeNi | AlCoCrFeNi | AlCoCrFeMnNi

A1 | B2 matrix (?) | A2 (?)

⇨ a) Effect of L-PBF process on microstructure and phase selection of HEA/CCAs
b) Precipitation behavior and its effects on strengthening mechanisms of HEA/CCAs

Outline

1 Introduction

2 Parameter developments of L-PBF process

3 Alloy developments

4 Nitride formation

5 Summary and future plans

2. Parameter developments of L-PBF process

(Flowability data from E. Gärtner)

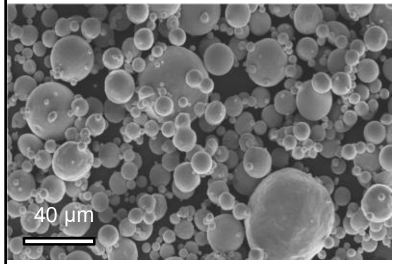
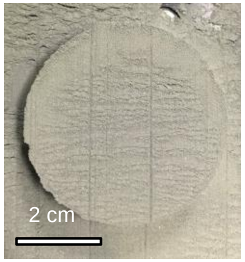
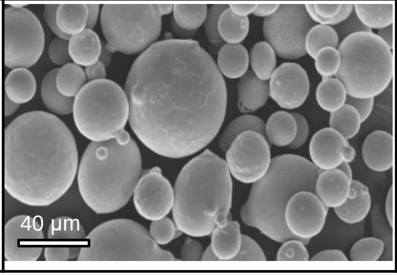
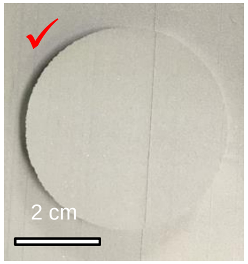
- Powder processability (CoCrFeNi alloy)

- Chemical composition (ICP-OES)

[at.%]

	Co	Cr	Fe	Ni
Powder	24.76	25.14	25.33	24.77

- Selection of particle size for L-PBF process

Powder	Image	Flowability: *CI*[1)] [%]	Flowability: *H*[2)]	Spreadability
0-90 µm		17.2	1.21	
		Fair level		
20-90 µm		14.5	1.17	
		Good level		

1) Compressibility index, CI

$$CI = \left(\frac{\rho_{tap} - \rho_{bulk}}{\rho_{tap}}\right) * 100$$

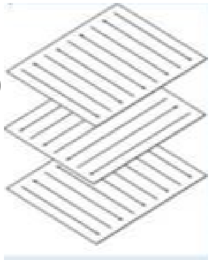
2) Hausner ratio, H

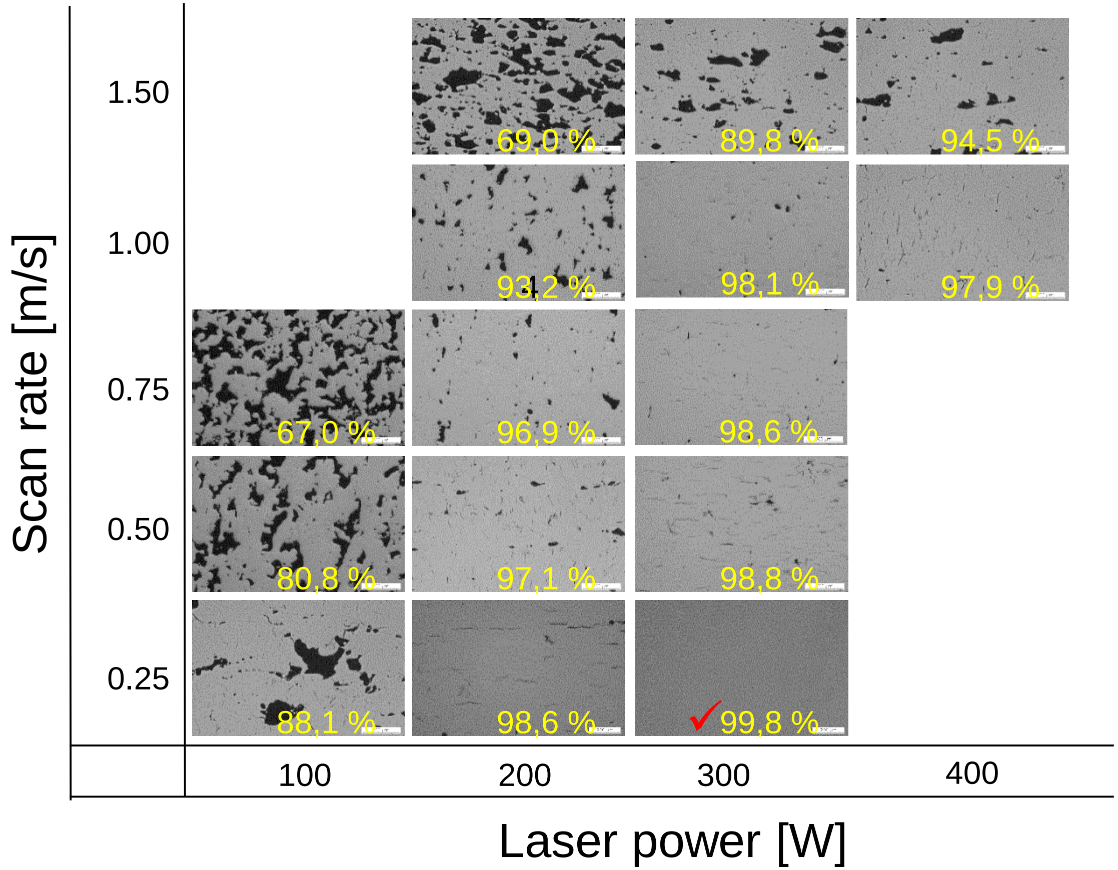
$$H = \frac{\rho_{tap}}{\rho_{bulk}}$$

⇨ Flowability and spreadability of HEA powder were improved by removal of fine particles. Powders with 20-90 µm was chosen for further parameter optimization.

- Parameter optimization of L-PBF process

- Raw materials: As-atomized powder of CoCrFeNi alloy (powder size: 20-90 µm)
- Part: Cube box (dimension: 8x8x8 mm^3)
- Hatching: Simple scanning pattern with 90° rotation



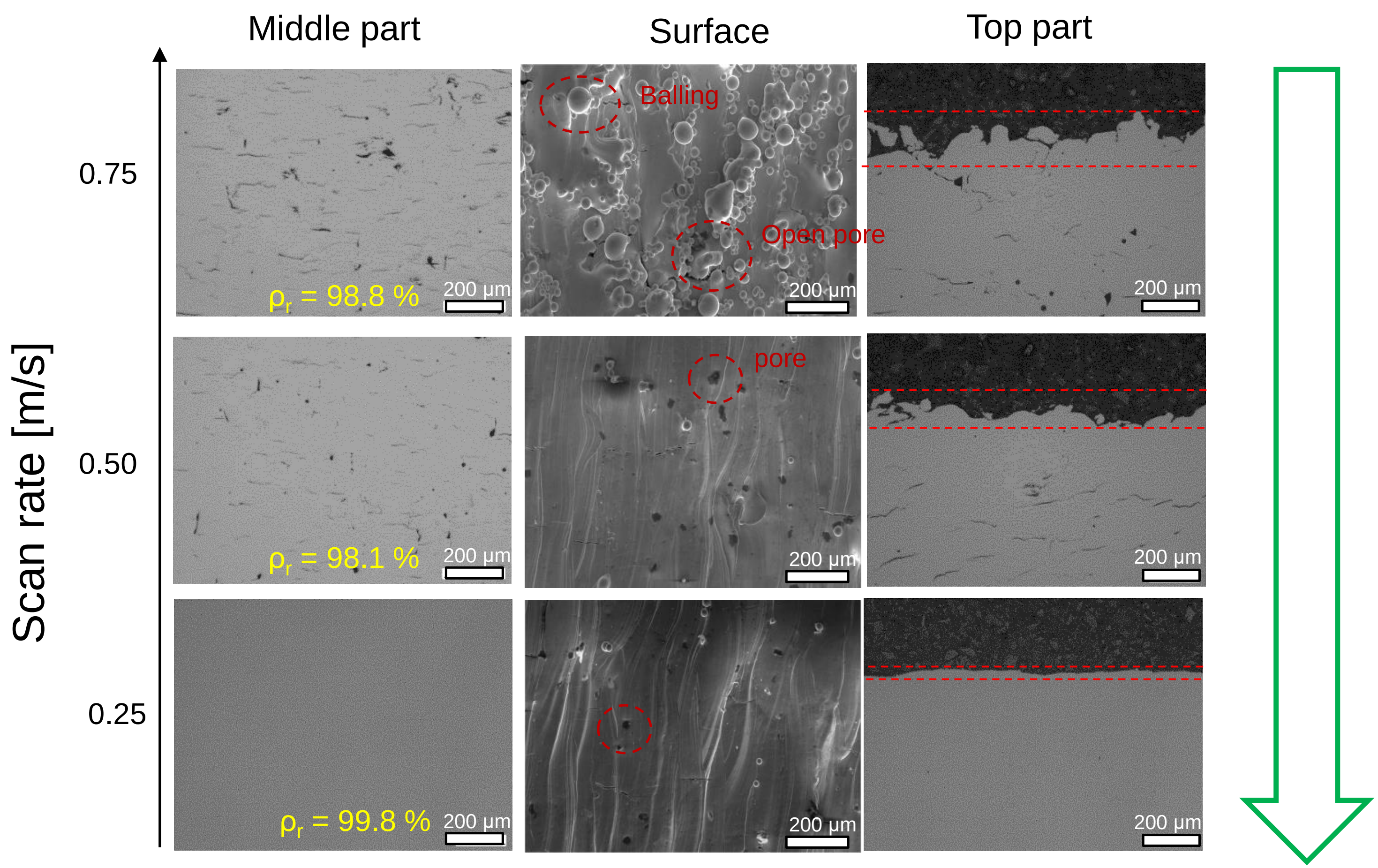


P [W]	Various
v [m/s]	Various
L [µm]	70
H [µm]	90
D [µm]	90

- Parameter optimization of L-PBF process

- As-built CoCrFeNi alloy fabricated by laser power of 300 W

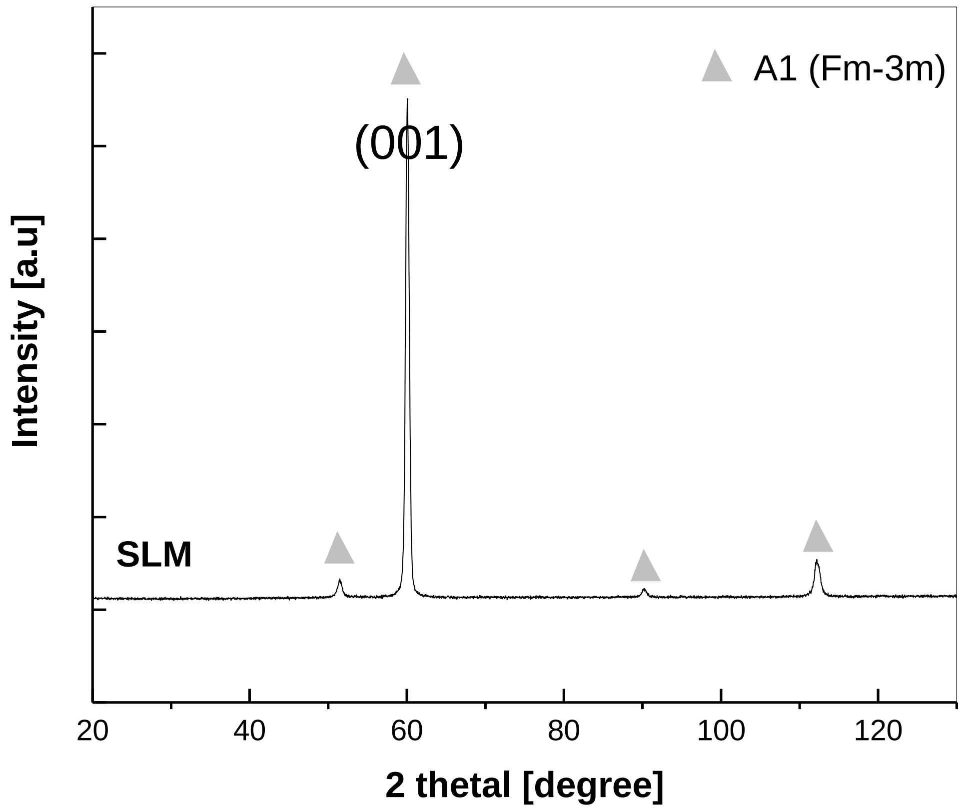
P [W]	300 W
v [m/s]	Various
L [µm]	70
H [µm]	90
D [µm]	90

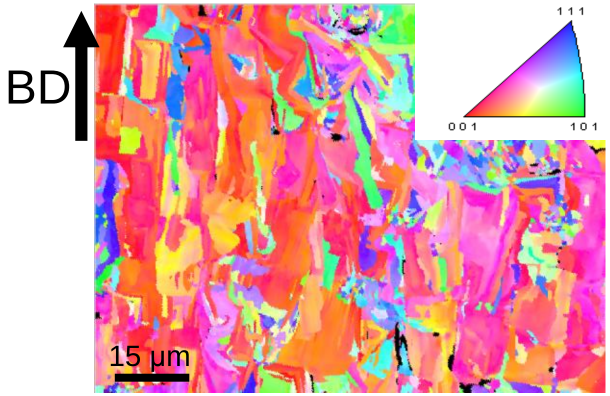


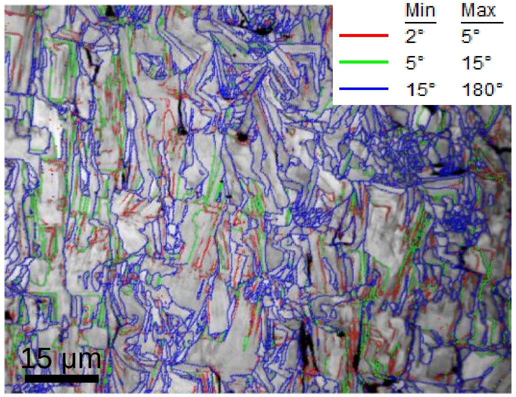
Sufficient melting and stabilized melt flow

- Microstructure evaluation of base alloy

- Phase analysis of CoCrFeNi alloy (*P* = 300 W, *v* = 0.25 m/s)





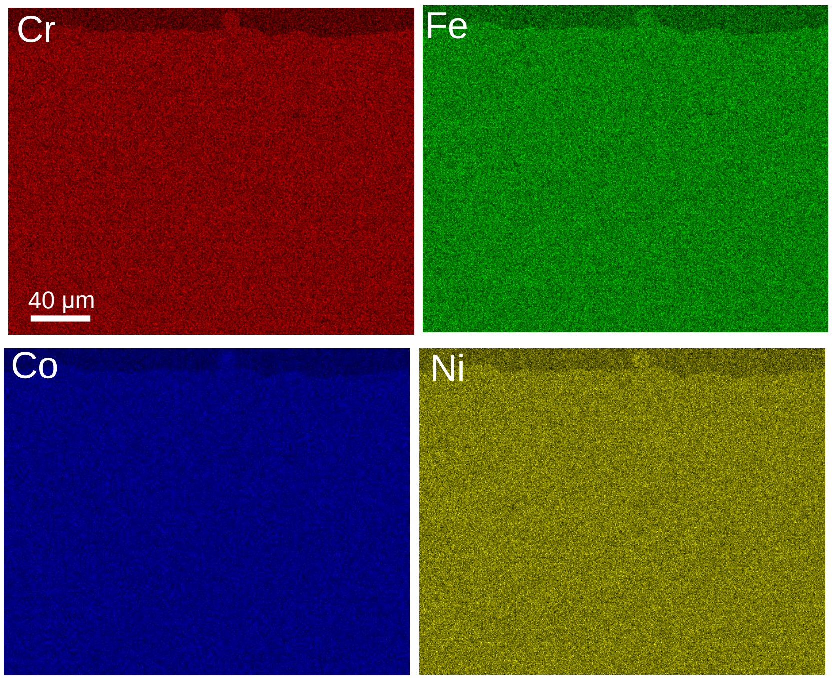


As-built CoCrFeNi alloy consists of single solid solution with A1 structure.
The alloy has a strong crystallographic texture with a preferential <100> growth along the building direction

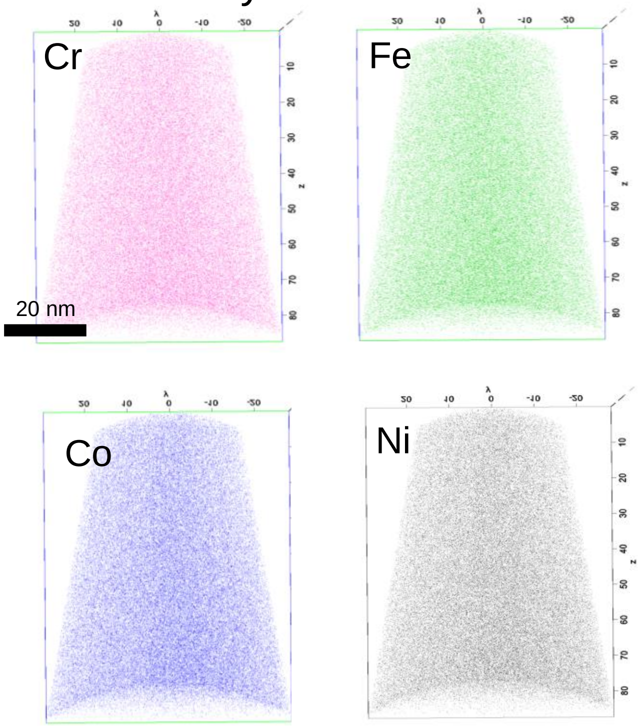
- Microstructure evaluation of base alloys

- Elemental distribution of CoCrFeNi alloy (P = 300 W, v = 0.25 m/s)

✓ SEM_EDS analysis



✓ APT analysis



⇨ The CoCrFeNi alloy has homogeneous atomic distribution in as-built condition

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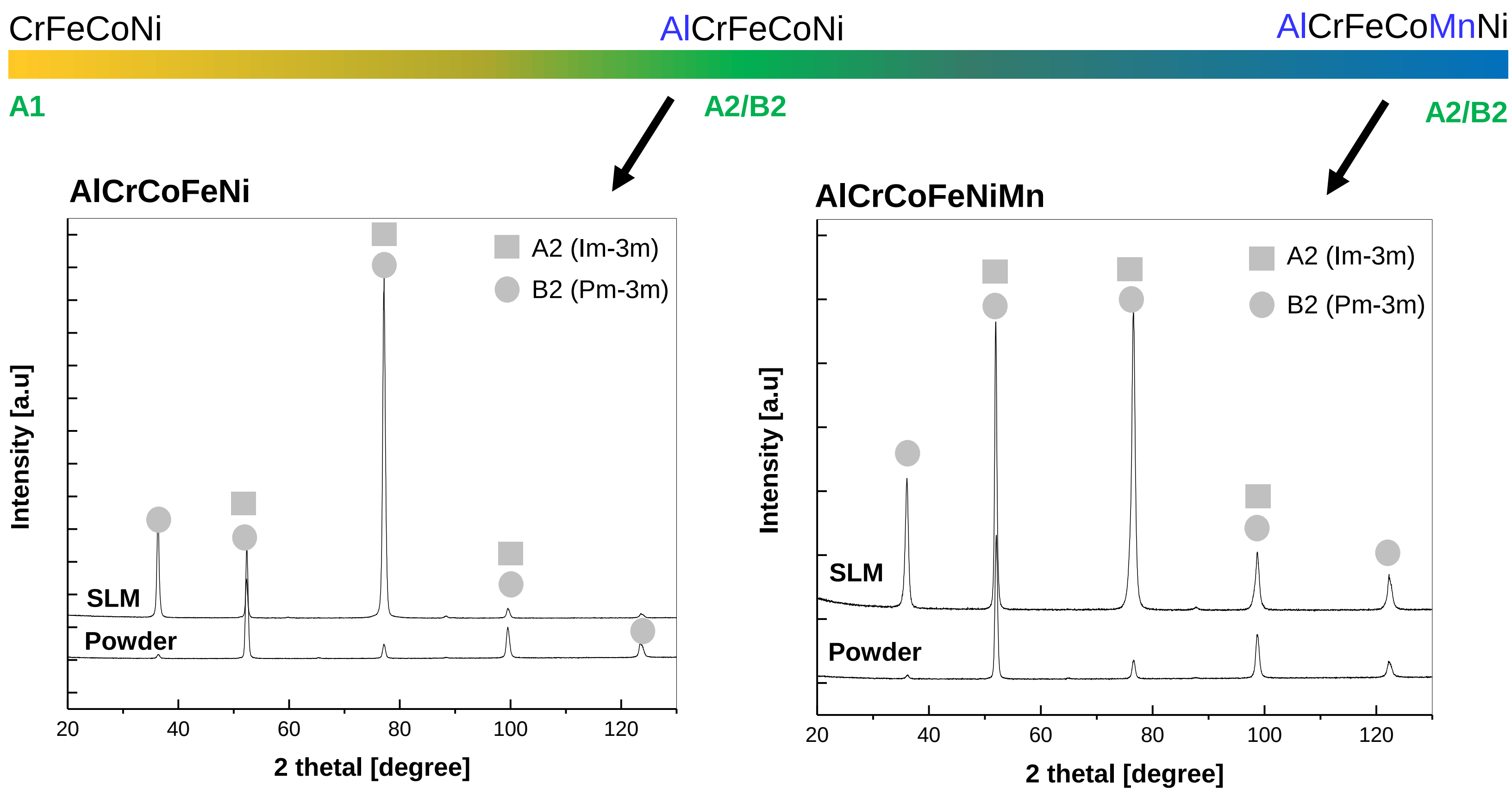
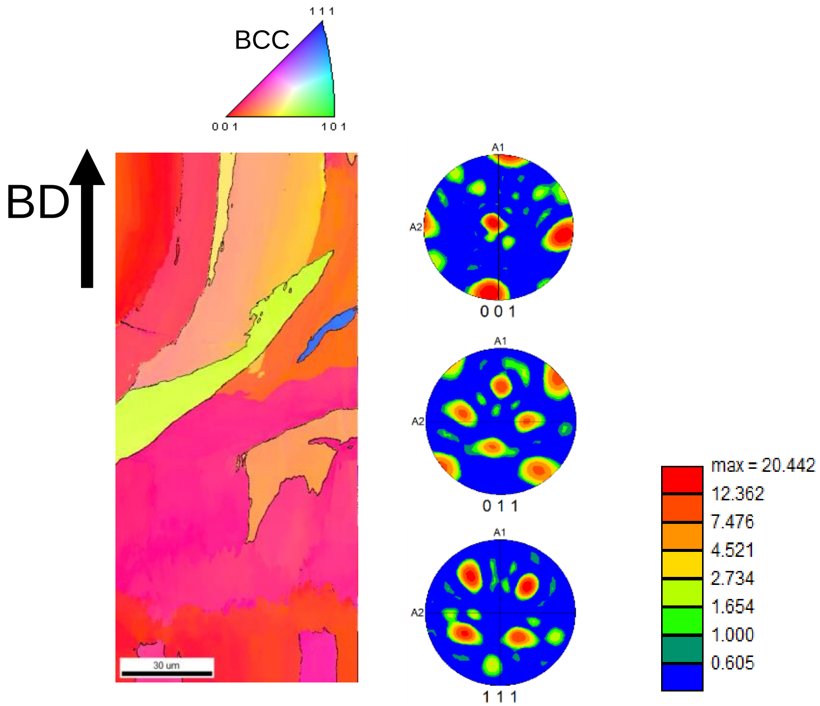
- Microstructure evaluation of base alloys



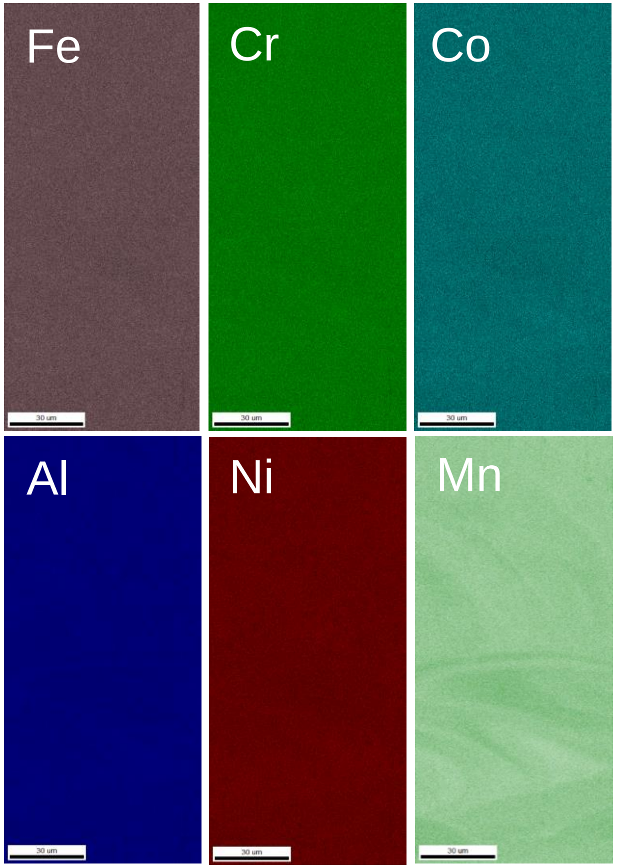
⇨ The AlCoCrFeNi(Mn) alloys consist of A2/B2 phase in XRD resolution

- Microstructure evaluation of base alloy

(a) IPF & PF map



(b) EDS map



As-built AlCoCrFeMnNI alloy has strong crystallographic texture, but no elemental segregation at grain boundaries

- Microstructure evaluation of base alloys

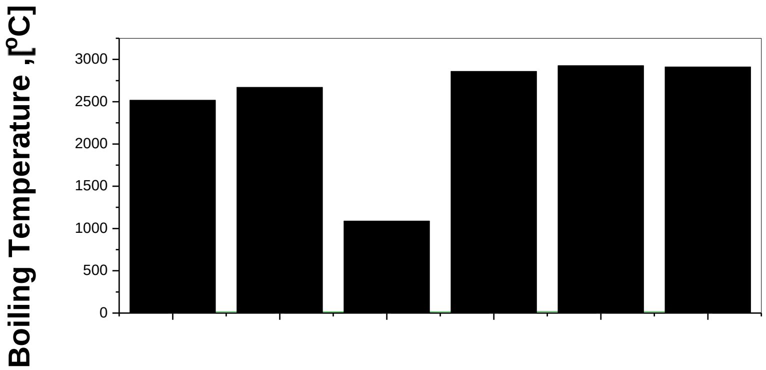
✓ Inhomogeneity of Mn in L-PBF samples

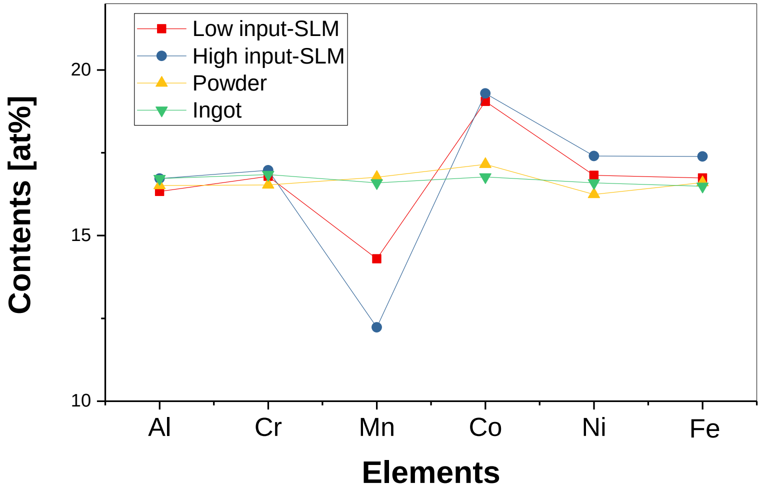
- Laser energy density (E) affects the temperature distribution of melt pool. The volume based energy density *E* (J/mm³) is defined as

$$E = \frac{P}{v * h * t}$$

E = Energy density (J/mm³), *P* = laser power (W), *v* = scan speed (mm/s), *h* = hatch spacing(mm), *t* = layer thickness(mm)

	L-PBF sample	
	Low E	High E
Scan rate [m/s]	0.50	0.25
Ev [J/mm³]	64	127



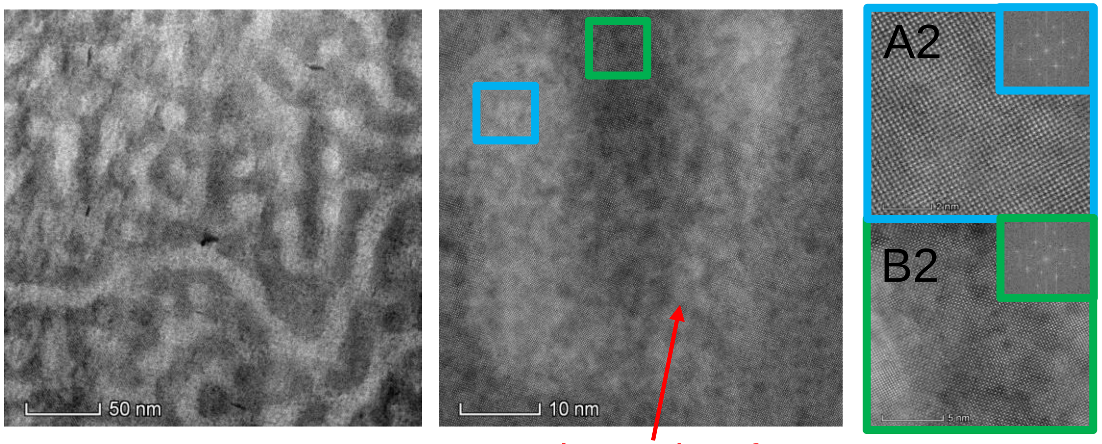


⇨ Owing to the low boiling temperature of Mn element, the L-PBF process involved Mn evaporation, which leads macro-scale elemental inhomogeneity of the alloys

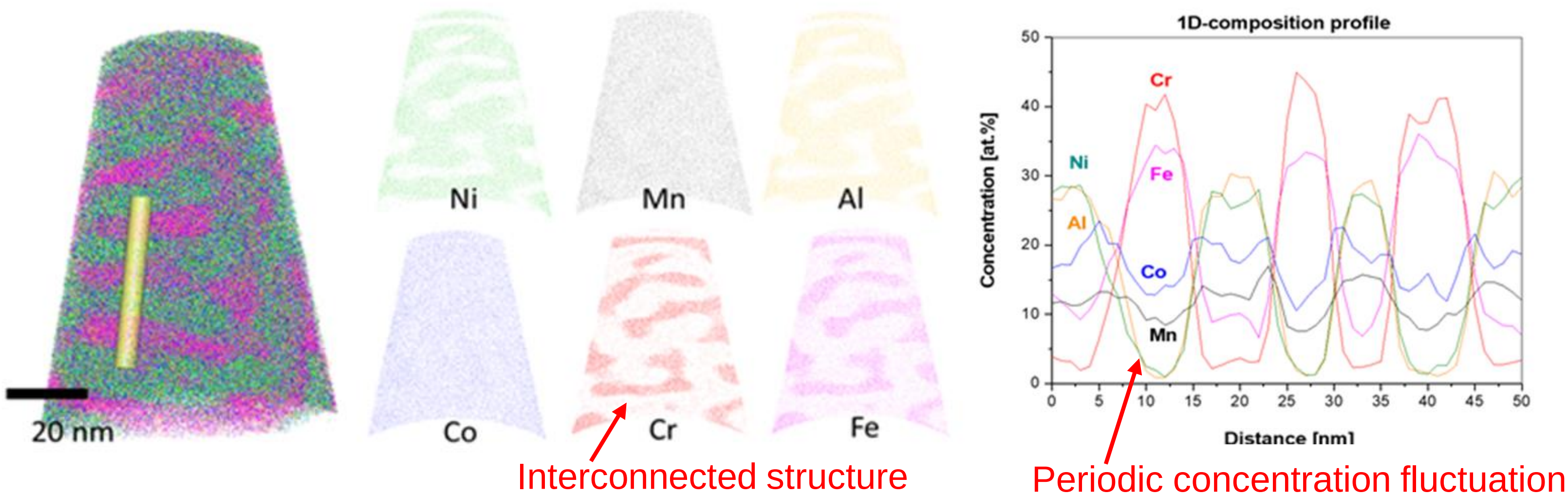
(TEM data from N. Peter)

- Nanostructure of AlCrFeCoMnNi alloy

(a) STEM-HADDF



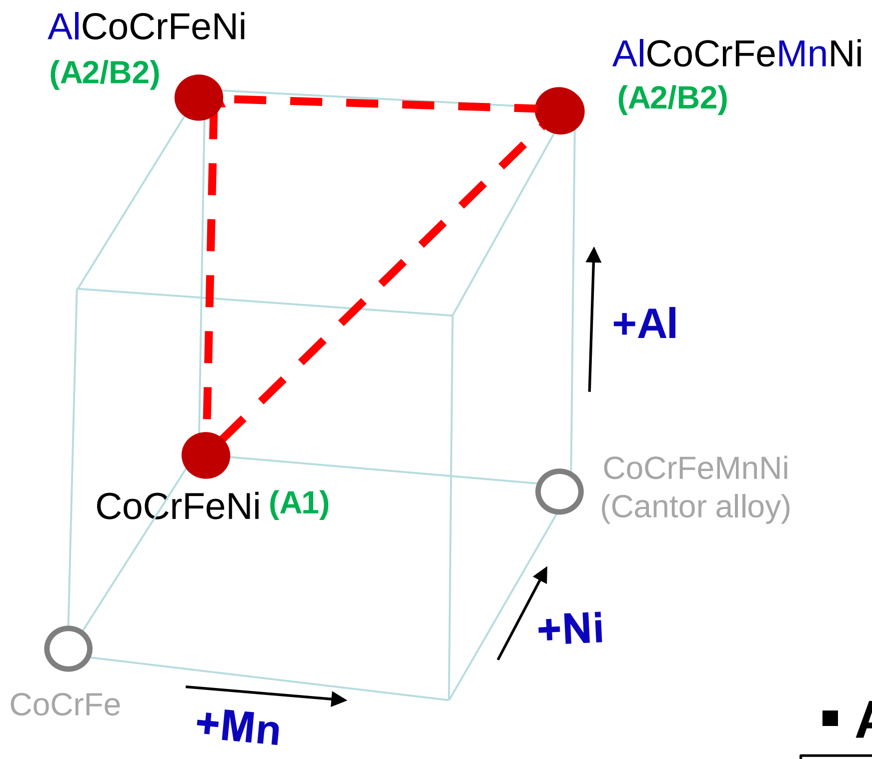
(b) APT reconstruction



The AlCoCrFeMnNi HEA has nano-scaled phase separation by spinodal decomposition

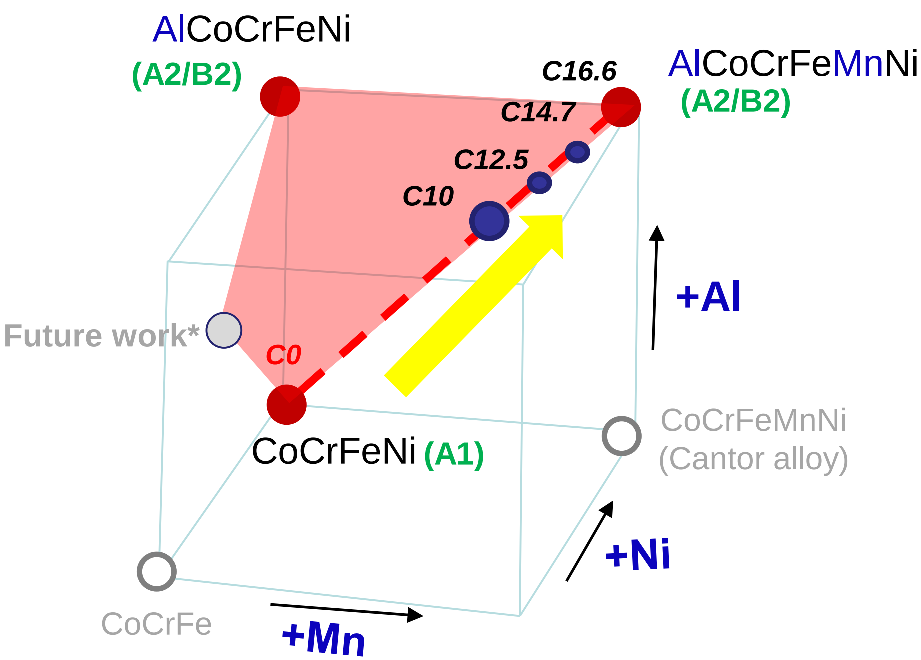
Outline

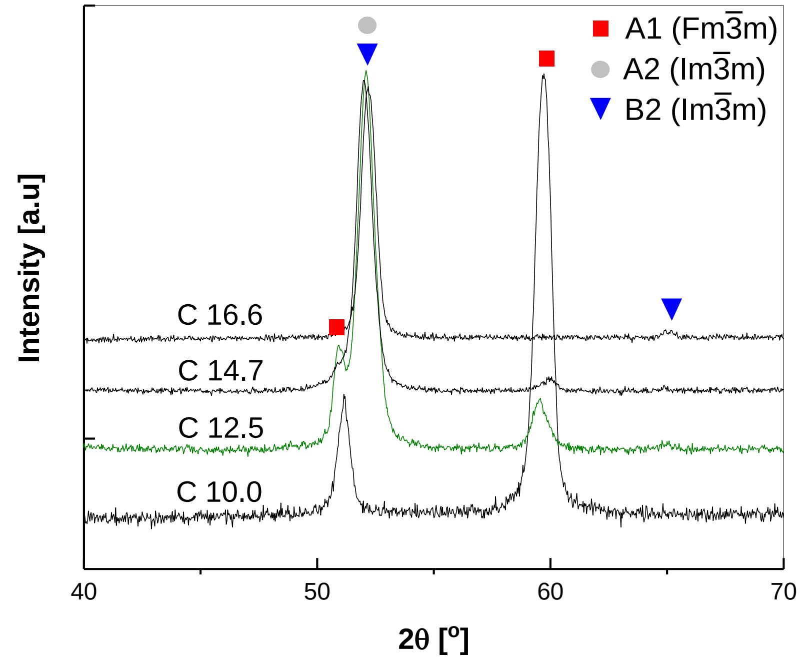
- **Alloys of interest**



- **Alloying method**

- **Alloys of interest**





Alloy	Composition	Phase
C16.6	$Al_{16.6}Co_{16.6}Cr_{16.6}Fe_{16.6}Mn_{16.6}Ni_{16.6}$	A2/B2
C14.7	$Al_{14.7}Co_{17.7}Cr_{17.7}Fe_{17.7}Mn_{14.7}Ni_{17.7}$	A1+A2/B2
C12.5	$Al_{12.5}Co_{18.8}Cr_{18.8}Fe_{18.8}Mn_{12.5}Ni_{18.8}$	A1+A2/B2
C10	$Al_{10}Co_{20}Cr_{20}Fe_{20}Mn_{10}Ni_{20}$	**A1**
C0	$Co_{25}Cr_{25}Fe_{25}Ni_{25}$	A1

⇨ The powder blending method was effective to design alloys forming desired phases by L-PBF process. The composition range of A1 phase was extended.

- Phase selection of HEA processed by L-PBF

✓ Conventional parameters for phase prediction in HEA

S. Guo et al. / Prog. Nat. Sci. 21 (2011) 433-446.

a) Valence electron concentration (VEC)

Y. Zhang et al. / Adv. Eng. Mater. 10 (2008) 534-538.

b) ΔH_{mix} (enthalpy of mixing) and δ (atomic radius difference)

c) CALPHAD, MD or Ab initio computational approaches

Phase prediction ≠ Experimental results (from L-PBF process)

✓ Reason for the expansion of A1 phase range

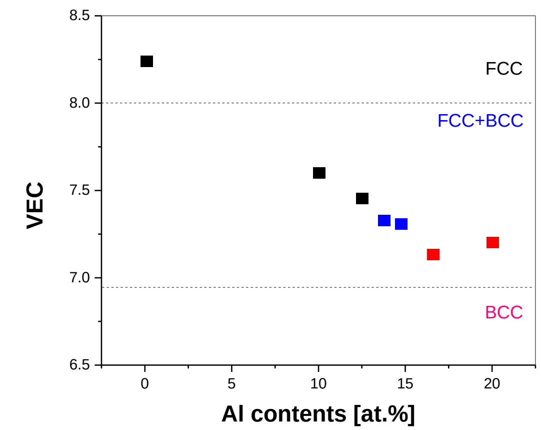
S. Guo et al. / J Alloys Compd 583 (2014) 410-413.

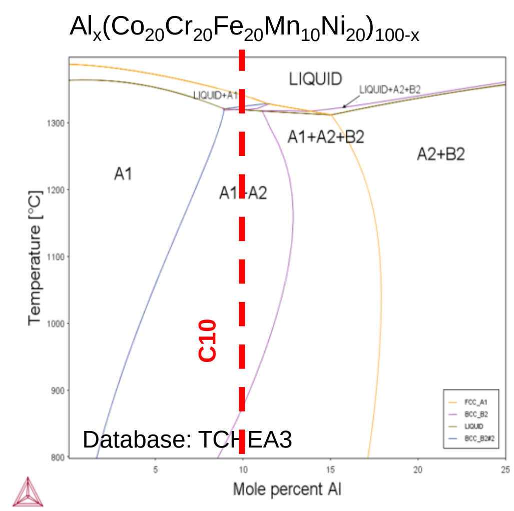
a) Al addition (leading lattice distortion and stabilization of BCC structure)

b) Rapid solidification rate (about 10^4~10^6 K/s) → solubility limits↑

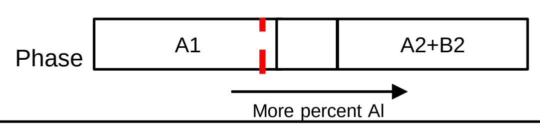
⇨ For the alloy development for or by L-PBF process, the rapid solidification effect should be considered.

Phase prediction (*C10* alloy : A1+A2)





Experimental results (*C10* alloy : A1)



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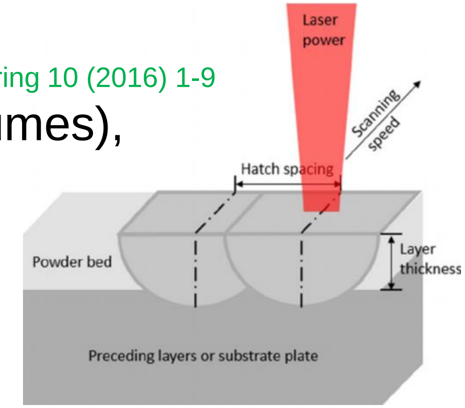
5 Summary and future plans

- Nitride formation during L-PBF process

- Role of gas flow in L-PBF process

  → Removal of process by-products (spatter and welding fumes), protection of oxide formation (in case of inert gas)

A. Ladewig et al. / Additive Manufacturing 10 (2016) 1-9



- Type of gas generally used in L-PBF process

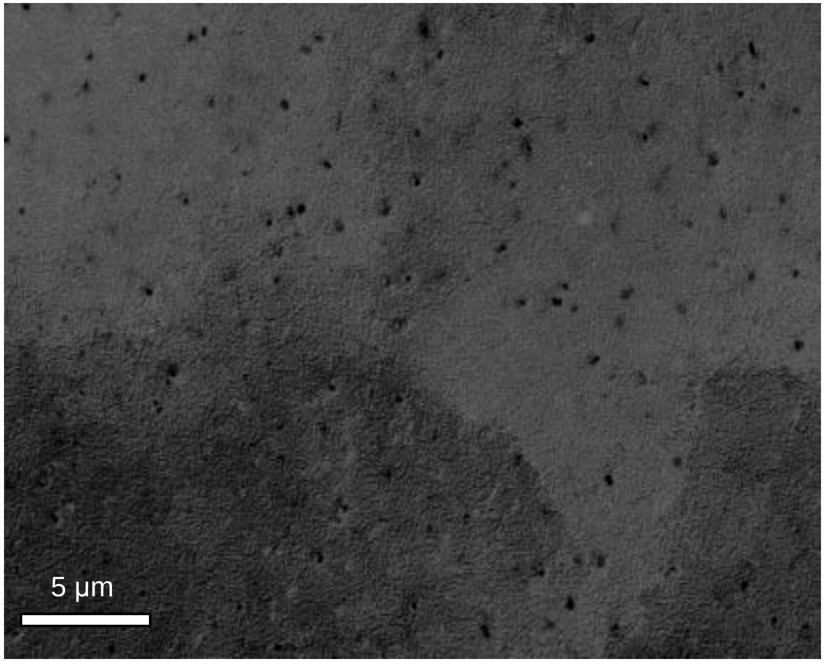
	Ar	N_2	He
Molar mass	40	28	4
The first ionization potential [eV]	15.68	14.54	24.46
Relative density of gas (air = 1)	1.38	0.97	0.14
Thermal conductivity [W/mK]	0.018	0.026	0.151

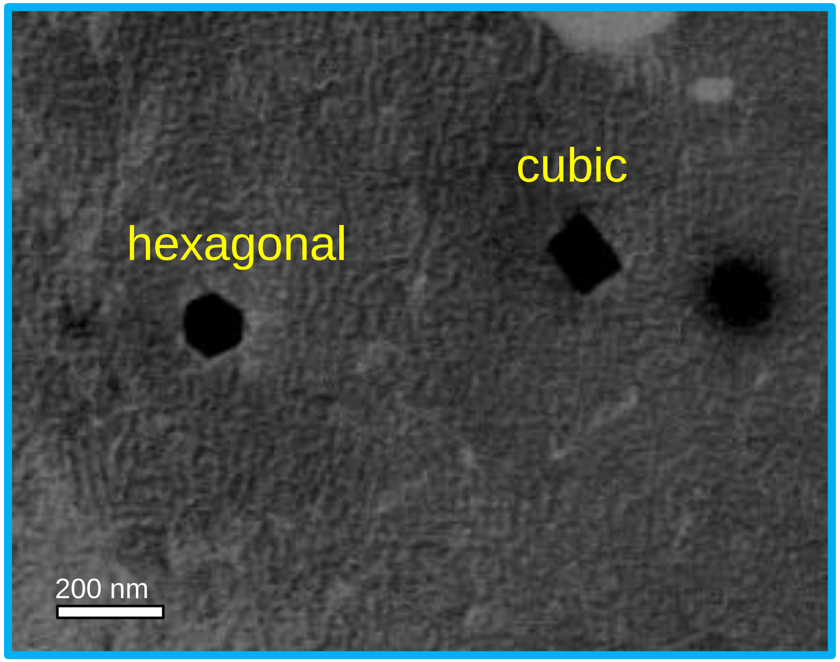
- Gas atmosphere effects plasma, penetration depth and melting behavior of the materials

⇨ Gas is important factor for L-PBF process and materials properties

- Nitride formation during L-PBF process

AlCoCrFeNiMn alloy (laser powder = 200 W, scan rate = 0.50 m/s)



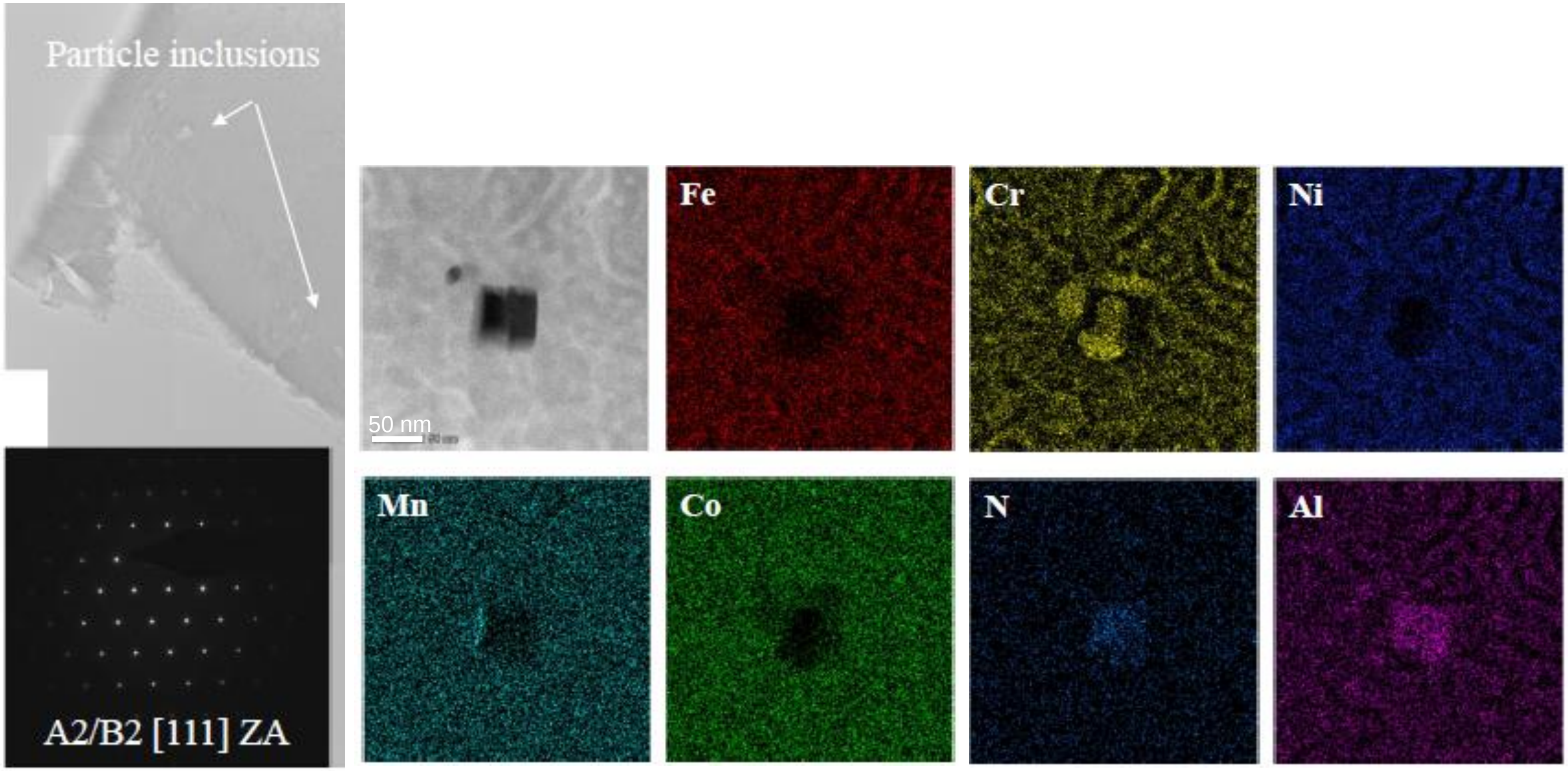


⇨ Using reactive N_2 gas flow during L-PBF process lead a formation of AlN particles

(TEM data from N. Peter)

- Nitride formation during L-PBF process

AlCoCrFeNiMn alloy (laser powder = 200 W, scan rate = 0.50 m/s)



⇨ L-PBF process under N_2 gas flow formed cubic AlN in A2/B2 matrix. The formation of cubic-nitride indicates the strong influence of rapid solidification rate of L-PBF process.

Summary

1. We investigated the influence of L-PBF process on micro- and nanostructure of CoCrFeNi and the Al-containing CoCrFeNi(Mn) alloy

   - Phase formation: CoCrFeNi alloy - A1 phase,
     Al-containing CoCrFeNi(Mn) alloy - A2/B2 phase
   - Nano-scaled modulated structure formed by spinodal decomposition

2. Alloy development method using powder blending and L-PBF process was effective to design HEAs consisted of desired phases

3. Nano-scaled nitrides were homogenously formed in AlCoCrFeNi(Mn) alloy by N_2 gas flow

Thank you for your attention!

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