

Process-microstructure-property relationships in Ni- and Fe-based alloys processed by AM



Alloys for Additive Manufacturing Workshop MPIE Düsseldorf

Prof. Dr.-Ing. Thomas Niendorf July 2016



Research Partners

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Institute of Materials Engineering

Agenda



- 1. Stainless steel 316L
- 2. Ni-based alloy IN 718
- 3. High-Mn Fe-based alloys





Agenda



1. Stainless steel 316L

- 2. Ni-based alloy IN 718
- 3. High-Mn Fe-based alloys







<u>Melting system:</u>	SLM 250 ^{HL} (SLM Solutions) • 400W fibre laser • Argon atmosphere	
Tested material:	 Stainless steel 316L Layer thickness: 30µm 	D
	 Average particle size: 40µm Platform temperature: 100°C 	D NA MITE

Treatment	1 (as-built)	2 (650°C)	3 (HIP)
Temperature [°C]	20	650	1150 (1000 bar)
Time [h]	-	2	4
Atmosphere	-	Argon	Argon

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Microstructure



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Tensile tests:

- in accordance with ISO6892-1:2009
- as-built surface
- displacement controlled (5mm/min)
- tests performed on testing machine Instron 5569
- at ambient conditions



condition	UTS / MPa	YS / MPa	ε _f / %
as-built/SLM surface	565 ±5 MPa	462 ±5 MPa	53.7 ±2.6 %
650 °C/turned surface	595 ±5 MPa	443 ±5 MPa	48.6 ±2.6 %
traditionally processed	530-680	220	≈ 40





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Fatigue tests:

- *in accordance with* ASTM E466-07
- as-built and turned surface
- force controlled
- Frequency: 40 Hz
- Stress ratio: R = -1

condition	fatigue limit $\overline{\sigma}$ [MPa]
as-built/SLM surface	108
as-built/turned surface	267
650 °C/turned surface	294
HIPed/turned surface	317
traditionally processed	240-381







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building direction

37,5 ±0,1

115

40 ±0,1

Rz 6,3

±0,03

5,1

37,5 ±0,1

9

/ Rz 25

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Woehler type S-N-curves



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Fracture mechanics tests: Comparison of threshold values

- Effect of post-treatments
- Effect of crack growth direction







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Higher threshold value for (\bot) -direction despite highest internal stresses in building direction superposed with testing load

=> small effect of internal stresses on crack growth

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Microstructure: Similar microstructure for as-built and 650°C

- elongated grains in building direction
- strongly textured

HIP condition exhibits

- · coarse grains, almost equiaxed
- absence of strong texture



as-built & 650°C

- Grains are elongated in the building direction
- Lower crack growth resistance along stretched grains => Lower threshold values
- For crack growth normal to building direction the boundaries are closer and act as barrier => higher threshold value



650°C

directior

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HIP

direction

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as-built

HIP

- Absence of preferred grain orientation leads to similar thresholds
- Increase in thresholds due to elevated grain size
- Grain boundary as barrier
- crack growth at low load levels stops dependent on the microstructure present in front of the crack tip =>higher scatter

	as-built	650°C	HIP
Threshold (⊥) [MPa⋅m¹/2]	4.3	-	4.7
Threshold (=) [MPa·m ^{1/2}]	3	3	4.6





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- 3. High-Mn Fe-based alloys







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What kind of microstructure can be obtained?

• Employed for processing of samples

→ SLM-280^{HL} → 400W / 1000W laser sources

- →layer thickness up to 150 µm
- → shell/core structures



Microstructure design

Microstructure/ grain shape

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Microstructure design

Microstructure/ grain shape

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Anisotropy/texture

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What kind of microstructure can be obtained?

- Microstructurally tailored
- Load-adapted design



Functionally graded by microstructure design



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Similar microstructure evolution for other alloys:

- > Solidification in cubic phase
- > No phase transformation upon cooling

➔ Ni-based alloys







Additive manufacturing is perfectly suited for direct microstructure manipulation

- Grain size & shape
- Anisotropy/ texture

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Presumably Nb-rich Laves phase

- Substructures in as-built condition
- Bright contrast in SEM => enrichment in Nb
- Laves phase particles



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	Solution heat treatment	Aging heat treatment	
C&W	980°C / 1.5h	720°C / 8h	620°C / 8h
SLM – as built	-	-	-
SLM – DA	-	720°C / 8h	620°C / 8h
SLM – 930°C	930°C / 1h	720°C / 8h	620°C / 8h
SLM – 1000°C	1000°C / 1h	720°C / 8h	620°C / 8h





- Substructures are thermally stable
- Solutionizing eliminates segregations





- Substructures are thermally stable
- γ'' -phase evolves upon ageing

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IN 718 – Creep



No significant difference between different orientations





■ SLM material → superior creep strength

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IN 718 – Creep







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IN 718 – Creep

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Compression creep



IN 718 – Creep

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Compression creep



3.5 % δ

5.8 % δ but
finer δ
subgrains
smaller γ"
depleted
zone

Conventional C&W material:

- δ phase is necessary to pin the grain boundaries during RX
- Less Nb is available for the precipitation strengthening phase γ''

SLM material:

- δ phase is not necessary
 - \rightarrow heat treatments can be adjusted
- More Nb is available for solid solution hardening and the formation of strengthening γ'' precipitates





Higher creep strength of SLM IN718

IN 718 – Fatigue

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Single laser 400 W



Similar to 316L

Post-treated conditions

Nomenclature	Condition	Details
S	Solution annealed	1000 °C/1 h/Air Cooling (AC)
Н	Hot isostatically	1150 °C/1000 bar/4 h/Furnace Cooling (FC)
	pressed (HIPed)	
S+A	Solution annealed	1000 °C/1 h/AC + 720 °C/8 h /FC at 50 °C/h to 621 °C
	+aged	+ 621 °C/8 h /AC
H+A	HIPed+Aged	HIPed + 720 °C/8 h/FC at 50 °C/h to 621 °C + 621 °C/8
		h/AC
P+H	Arc-PVD+HIPed	1000 °C/1 h/AC + Arc-PVD(Ni-20Cr) + HIPed
P+H+A	Arc-	1000 °C/1 h/AC + Arc-PVD(Ni-20Cr) + HIPed +
	PVD+HIPed+Aged	720°C/8 h/FC at 50 °C/h to 621 °C + 621 °C/8 h/AC

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Optical microscopy







Low degree of porosity \rightarrow BUT: local differences

→ Most critical: porosity close to the surface cannot be eliminated by HIP

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IN 718 – Fatigue

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IN 718 – Fatigue





Key parameter:

- Plastic strain
- Mean stress



Energy dissipation per cycle strongly increases upon HIP Microstructure design in Ni-based alloys

→ Thermal stability during solutionizing?





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→ EBSD: Hardly any change in microstructure up to 1000 °C
 → <u>BUT</u>: Significant changes in hardness

Microstructure on different length scales

- → Sub-structures
- → Laves-Phase
 - Partially dissolved upon solutionizing at 1000 °C





15 µn

V

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[122]

15 µm

BD

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Microstructure on different length scales

- ➔ Sub-structures
- → Laves-Phase
 - ➔ Partially dissolved upon solutionizing at 1000 °C
- \rightarrow γ "-phase evolves upon aging

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Tensile tests: In good agreement with hardness evolution





Compression creep:

- ➔ Laves-Phase detrimentally affects creep rate
- → Inferior to the fine grained condition

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3. High-Mn Fe-based alloys





300 µm



300 µm

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New Materials processed by Additive Manufacturing

- Fe-based alloys
- TWIP steel
 - → delayed necking due to twinning
 - → extreme ductility
 - ➔ high hardening capability



Main goals

- Determination of mechanical performance and microstructural characteristics
 - after SLM
 - after heat treatment
- \rightarrow Comparison to thermo-mechanically processed blanks

Powder processing

→ TLS Technik, Bitterfeld

Process parameters

→ Same as for 316L stainless steel

Heat treatment

(aiming in recrystallization)

- In vacuum atmosphere
- 1h, 1050°C

Specimen geometry



Bulk material

Microstructure – Scanning electron microscopy



- Fairly large grains (Reference material from blank: 2-5 µm)
- Grains elongated parallel to BD
- Parallel features following deformation in numerous grains → Twins?
- Dislocation cells in several grains
 - ➔ submicron-scale
- Following heat treatment
 → large equiaxed grains



Microstructure – Scanning electron microscopy -EBSD



- Orientations plotted for LD
- Weak local texture for Asbuilt
- Following deformation
 → <111> <001> texture
- Slip and twinning
 → TWIP effect

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Microstructure – X-ray diffraction



- BD dominated by <001> in as-built
- <001> <111> in LD following deformation
 - → Slip and twinning → TWIP
- Randomization following heat treatment



Mechanical properties

suffer from surface quality

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- Reference: Conventionally processed sheet; UTS: 1160 MPa Elongation to failure: ~50 %
- Not ground SLM processed TWIP steel

→ slightly lower values

- SLM processed + ground
 almost equal
- Further increase of ductility following heat treatment



Prof. Dr.-Ing. Thomas Niendorf T. Niendorf et al., Mater. Character. 85, 2013, 57-63.

gauge

measured by use of caliper

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Mechanical properties

- Dimple like fracture → high ductility
- TWIP steel does not severely suffer pores



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Microstructure:

- \rightarrow elongated grain morphology
- \rightarrow micro-scaled substructures
- \rightarrow <001> texture for BD

Mechanical performance:

→ High strength and ductility already in the as-built condition
→ TWIP effect

Compared to thermo-mechanically processed material:

 \rightarrow Similar properties

Well suited base material for future work

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SLM TWIP steel as base for a new alloy solely processable by AM



TWIP-Ag

Bioresorbable implants

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Microstructure – SEM

→ Ag is fairly homogeneously distributed within the TWIP matrix



New materials

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Impact on the global and local corrosion behavior are evident
 Degradation rates in the human body should be increased

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Conventionally processed Fe-Mn-Al-Ni SMAs

- > α (BCC) γ (FCC) phase transformation
- Max. transformation strain 12 % ([123] oriented single crystal)
- Theoretical transformation strains:
 26.5 % (Tension, [001])



Ref.: Omori et al., Science 2011

Conventionally processed Fe-Mn-Al-Ni SMAs

 β -precipitate: B2 (NiAl)

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Precipitation of β-phase particles induced by ageing at 200°C
 Pre-requisite for thermo-elastic martensitic transformation

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Ref.: Omori et al., APL 2012



HR-TEM: Nano-scaled β -phase precipitates

Conventionally processed Fe-Mn-Al-Ni SMAs

- > Low CC-slope (Increase of transformation stress at increasing temperature)
- ➢ Pseudoelastic temperature window 350 K
 - \Rightarrow Numerous fields for application



Critical transformation stresses for PE-response as a function of temperature for diverse SMAs



Application temperature ranges of diverse SMAs

Ref.: Omori et al., Science 2011

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Conventionally processed Fe-Mn-Al-Ni SMAs

Impact of microstructure



Stress (MPa)

Stress (MPa)

Fe-Mn-Al-Ni, Impact of grain size

Ref.: Omori et al., APL Materials 2013

Pronounced texture and coarse
microstructure are needed
→ Minimization of grain constraints



PE response of Fe-Mn-AI-Ni wires in different conditions

Fe-based shape memory alloys

Fe-Mn-Al-Ni SMAs processed by SLM

- Solidification in cubic phase (bcc)
- Similar impact of processing parameters and geometry as in case of 316L

Direct microstructure design

➢ BUT: Surface cracking



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Fe-based shape memory alloys

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 BUT: High driving force for abnormal grain growth
 bamboo structures

Fe-based shape memory alloys

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N I K A S S E L

Fe-Mn-Al-Ni SMAs processed by SLM



BUT: Rapid functional degradation

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Acknowledgements:

- DFG and DMRC for funding
- Dr. D. Schwarze (SLM Solutions)
- E. Aydinöz, F. Brenne, P. Kanagarajah, Dr. S. Leuders, A. Riemer (University of Paderborn, DMRC)
- Dr. S. Neumeier, M. Pröbstle (Materials Science & Engineering, Institute I, FAU)

Questions?


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Inconel 939

- Ni-based superalloy
- Typical material for high-temperature applications

Main goals

- Determination of microstructural characteristics
 - after SLM
 - after solution-annealing
 - after ageing



Source: www.schleifblog.de

- Examination of mechanical properties
 - cyclic / montonic
 - room temperature / 750°C
 - horizontally / vertically manufactured specimens
- → Comparison to conventionally fabricated Inconel 939

Process parameters

	volume contour	volume area
hatch distance, mm	0.15	0.12
laser power, J s ⁻¹	100	175
laser speed, mm s ⁻¹	540	620
layer thickness, µm	30	30

Heat treatment

(standard for land-based turbine blades)

- In vacuum atmosphere
- Solution annealing: 4h, 1160°C
- Single stage ageing: 16h, 850°C

Specimen geometry



 Local chemical compositions



Microstructure, as-built – optical microscopy





→ Arch-shaped lines resulting from melt pool

Perpendicular to building direction

 \rightarrow Elongated structure due to laser movement

Microstructure, as-built – electron backscatter diffraction







- Columnar grains alongside the building direction
- Grains grow across several layers (>30µm)
- Thicker and shorter grains in case of vertically manufactured sample due to additional heat flux towards grip sections

→ No relation between the features as visible by optical microscopy and the grain orientation and morphology

Microstructure, aged – electron backscatter diffraction









ue to high annealing temperatures, high residual stresses)

ening → In comparison to cast material still small grain sizes!

of vertically manufactured sample (average grain diameter 35 μm vs. 70 μm)

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Microstructure – X-ray diffraction



• Preferred orientation of the [001] fibre alongside BD \rightarrow optimally suited for high temperature creep

 \rightarrow Grain orientation highly dependent on heat flux during processing

• Much weaker texture after ageing due to recrystallization



As-built microstructure – transmission electron microscopy



- Elongated structures on very small scale parrallel to the building direction
- Likewise cell-shaped strucutures were found
 - \rightarrow Formation and distribution still is unclear
 - → Effect on the mechanical properties...?!



Microstructure, aged – transmission electron microscopy



- γ '-precipitates (Ni...)₃(AI,Ti,...) in both after SLM and casting
 - \rightarrow Hardening effect
 - \rightarrow Resistance against high temperature creep
- γ '-precipitates are larger in case of SLMed material \rightarrow reason?

Microstructure – transmission electron microscopy





- In addition to γ'-precipitates brittle phases are present already after ageing (normally after long time operation)
- Smaller γ'-precipitates already in the solution annealed condition (normally first during ageing)

 \rightarrow Formation kinetics much faster than in cast material

 \rightarrow Nuclei for precipitations are formed during SLM

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Influence of building direction



Loading axis alongside building direction:

- Higher ductility
- Yield strength almost equal

Microstructural reasons:

- Cell-shaped substructures act as barriers for dislocation movement \rightarrow similar σ_v
- Elongated grains alongside BD → higher ductility

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Monotonic mechanical properties – impact of heat treatment



 \rightarrow Overall increase of yield strength

Microstructural reason: Formation of precipitates \rightarrow overall increase of σ_v

Loading axis alongside building direction:

- Higher yield strenght
- Ductility equal

Microstructural reason:

Substructures get dissolved, yield strength dominated by grain size \rightarrow higher σ_v

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Monotonic mechanical properties – influence of temperature



Clearly decreased ductility of as-built condition

 \rightarrow Fast formation of precipitates + embrittlement

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Monotonic mechanical properties – comparison to cast material



 $\sigma_{y,SLM}$ = 750 MPa – 950 Mpa $\sigma_{y,cast}$ = 500 MPa – 800 Mpa

as-built / as-cast - RT:

- \rightarrow Significantly higher ductility in case of as-built SLM

60 µm

300 µm

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Temperature influence – as-built / as-cast conditions



 $\sigma_{y,SLM}$ = 750 MPa – 950 Mpa $\sigma_{y,cast}$ = 500 MPa – 800 Mpa

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Temperature influence – aged condition



σ_{y,SLM}= 750 MPa – 950 Mpa σ_{y,cast}= 500 MPa – 800 Mpa

Fatigue lifes - as-built / as-cast condition

condition	SLMed, as-built	SLMed, aged	cast, as-cast	cast, aged
N _f , RT	4702	1598	313	2677
N _f , 750 °С	209	73	230	272

→ At RT higher fatigue life for as-built SLMed material (although not polished)

Reason:

Strain amplitude of 0.5 % leads to relatively high stresses

- \rightarrow Yield strength of cast material is exceeded
- \rightarrow Loading of SLMed material is mere elastic

At lower strain amplitudes (0.35 %) similar fatigue lifes were reached for both SLM and cast material



Fatigue lifes – aged conditions

condition	SLMed, as-built	SLMed, aged	cast, as-cast	cast, aged
N _f , RT	4702	1598	313	2677
N _f , 750 °С	209	73	230	272

Reason:

- SLM: Formation of brittle phases and process induced defects (pores)
- \rightarrow Higher sensitivity to crack initiation and growth

Cast: Increase of yield strength due to precipitates \rightarrow Lower plastic strain amplitude

→ Lower fatigue life for aged SLMed material (even if polished)





Fatigue lifes – temperature influence

condition	SLMed, as-built	SLMed, aged	cast, as-cast	cast, aged
N _f , RT	4702	1598	313	2677
N _f , 750 °С	209	73	230	272

→ General reduction of fatigue life in comparison to RT

Reason:

Enhanced dislocation mobility, typically occurring at high temperatures

Restricitions:

Only few tests were conducted \rightarrow scatter?

Conclusions

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Microstructure:

- \rightarrow fine columnar grains
- \rightarrow micro-scaled substructures

Two-stage heat treatment:

- \rightarrow recrystallization
- \rightarrow still grain sizes significantly lower than in the cast alloy

Monotonic load:

- \rightarrow High ductility of the as-built condition
- \rightarrow Increase of yield strength and embrittlement by ageing

Compared to cast material:

→ always higher yield strength in case of SLMed material







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Cyclic loading at RT:

 \rightarrow Better performance of the as-built SLM-processed condition





- \rightarrow fast formation of precipitates
- \rightarrow embrittlement of the SLM-processed as-built condition
- ightarrow Reduction of ductility and fatigue lives

