Additive Manufacturing of Metals and Alloys

8. Post-treatments for microstructure and properties control

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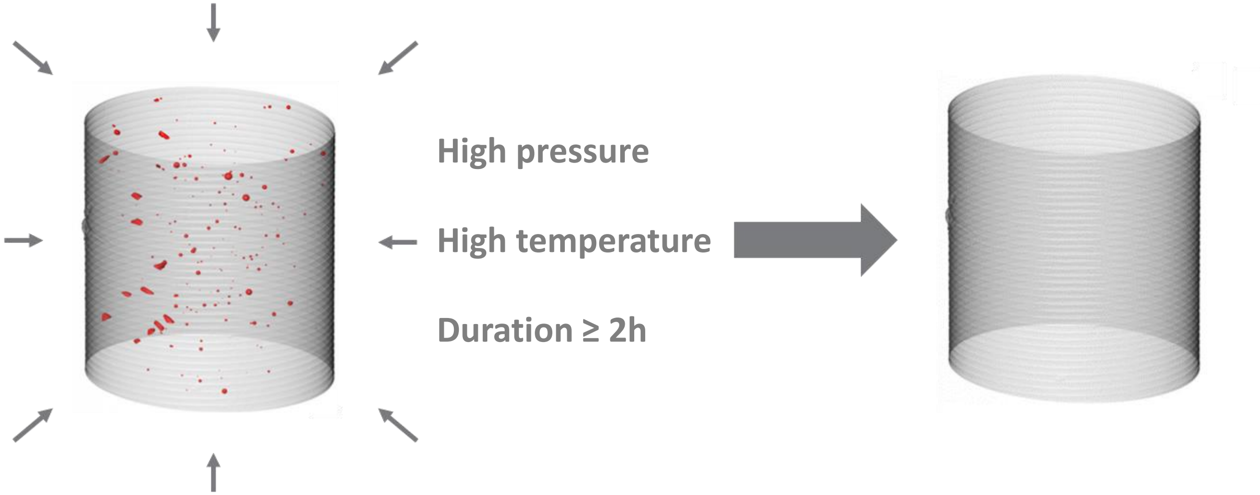
Post-treatments for microstructure and properties control

- HIP
  - process conditions
  - limitations
  - microstructural effects
- Stress-relief
  - process conditions
  - microstructural effects
- Thermal post-treatments for microstructure control
  - typical microstructural effects
  - influencing factors (illustrated on Ti-6Al-4V)
- Surface post-treatments
- Effect of post-processing on mechanical properties
  - tensile
  - fatigue

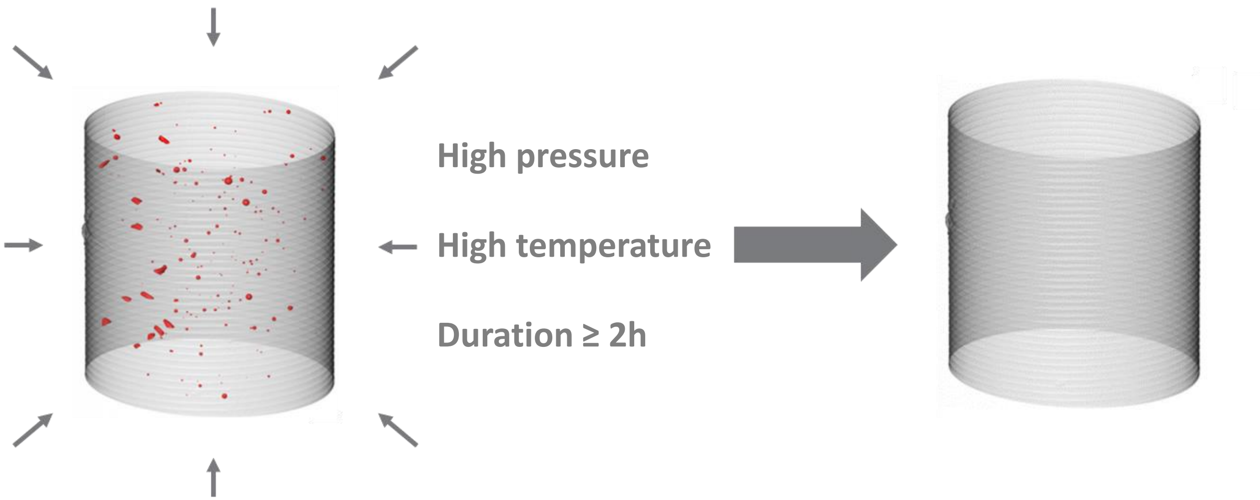
Hot Isostatic Pressing (HIP)



HIP is a thermomechanical post-treatment allowing to remove pores entrapped in the as-built AM material.

Post-HIP relative densities **typically exceed 99.9%.**

HIP: process conditions



HIP parameters (duration, temperature, pressure) are **material-dependent.**

Material		Temperature	Pressure	Duration
Ni-based alloys	IN718	1200°C	120 MPa	4h
		1180°C	150 MPa	3h
		1150°C	100 MPa	4h
	IN625	1120–1240 °C	100–165 MPa	3-4h
	Hastelloy X	1155°C	100 MPa	3h
Ti alloys	Ti-6Al-4V	900°C – 920°C	100 MPa	2h
Al alloys	AlSi10Mg	500°C	100 MPa	2h
Mg alloys	AZ61	450°C	103 MPa	3h
Stainless steel	316L	1125°C	137 MPa	4h
		1150°C	100 MPa	4h
	17-4 PH	1120°C	105 MPa	2h
CoCr alloys	CoCrMo	1200°C	100 MPa	4h

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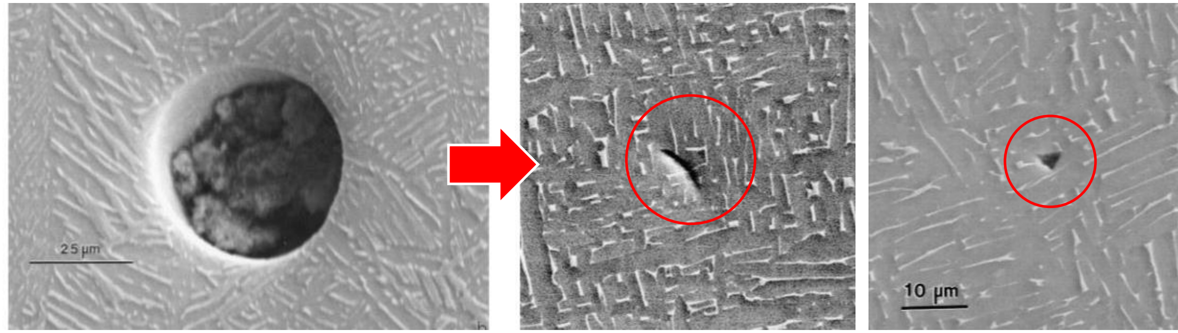
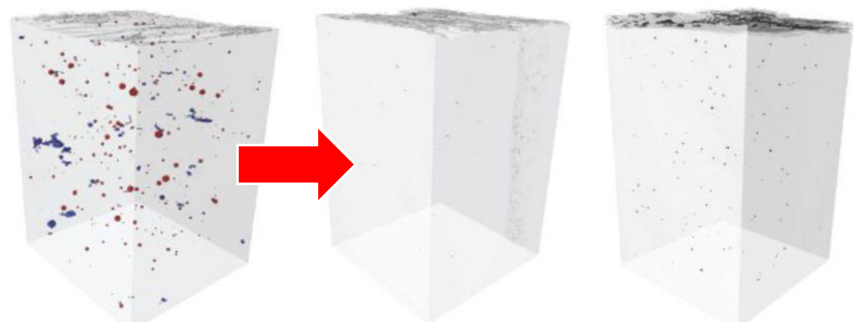
HIP: limitations

Remnants = small argon-filled collapsed voids.

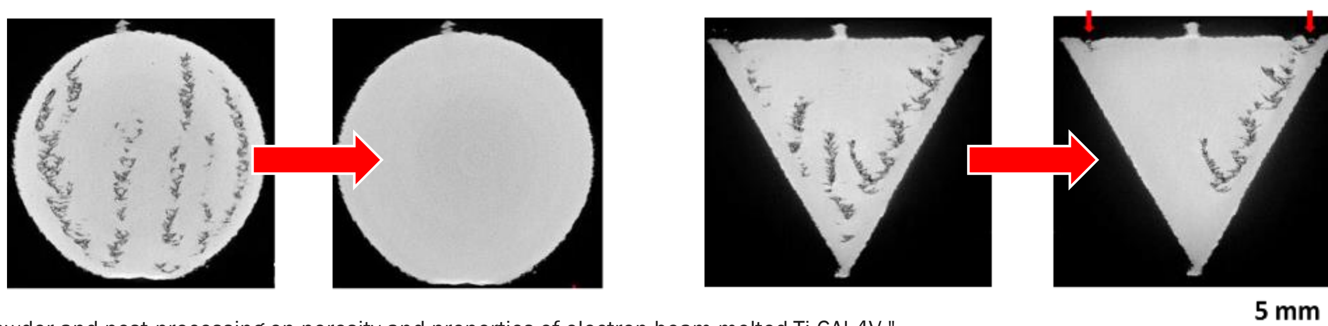


HIP induces a radius shrinkage of 1 to 2 orders of magnitude, resulting in a modest to significant increase in the internal pressure of the Ar-containing pores, which impairs their full closure.

Regrowth



Ineffective for open porosity



Cunningham, Ross, et al. "Analyzing the effects of powder and post-processing on porosity and properties of electron beam melted Ti-6Al-4V." *Materials Research Letters* 5.7 (2017): 516-525.
Tammas-Williams, Samuel, et al. "The effectiveness of hot isostatic pressing for closing porosity in titanium parts manufactured by selective electron beam melting." *Metallurgical and Materials Transactions A* 47.5 (2016): 1939-1946.

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HIP: microstructural effect

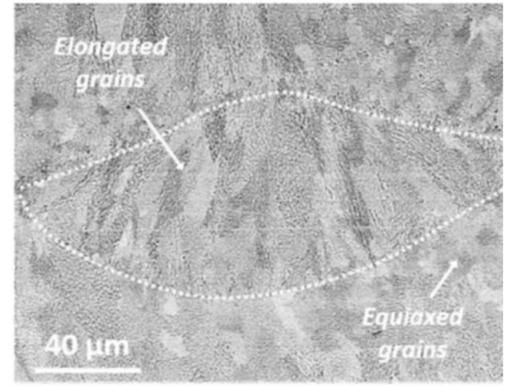
Al-based alloys (AlSi10Mg)

Melt pool boundaries are no longer visible after HIP.

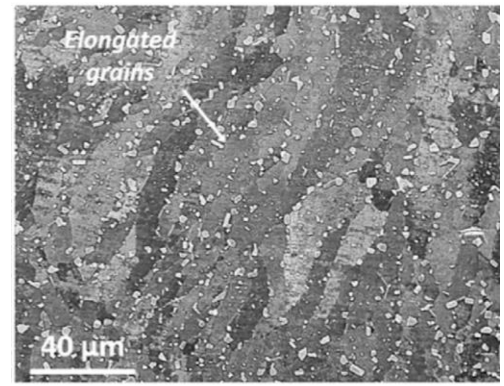
<u>Prior to HIP</u>:
fine, sub-micron cellular eutectic microstructure, elongated along the build direction

<u>Post HIP</u>:
diffusion and agglomeration of Si into large Si particles

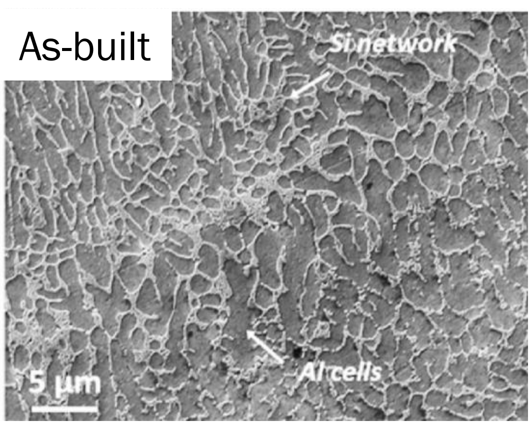
As-built



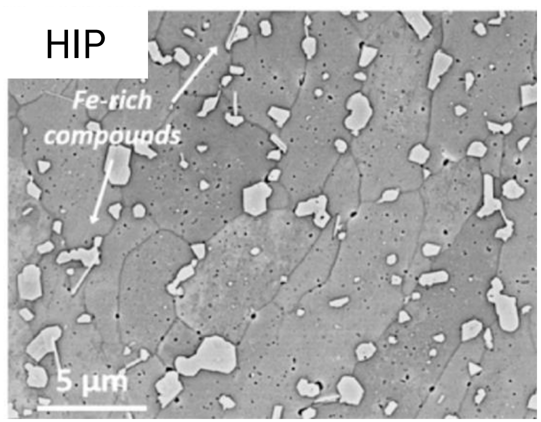
HIP



As-built

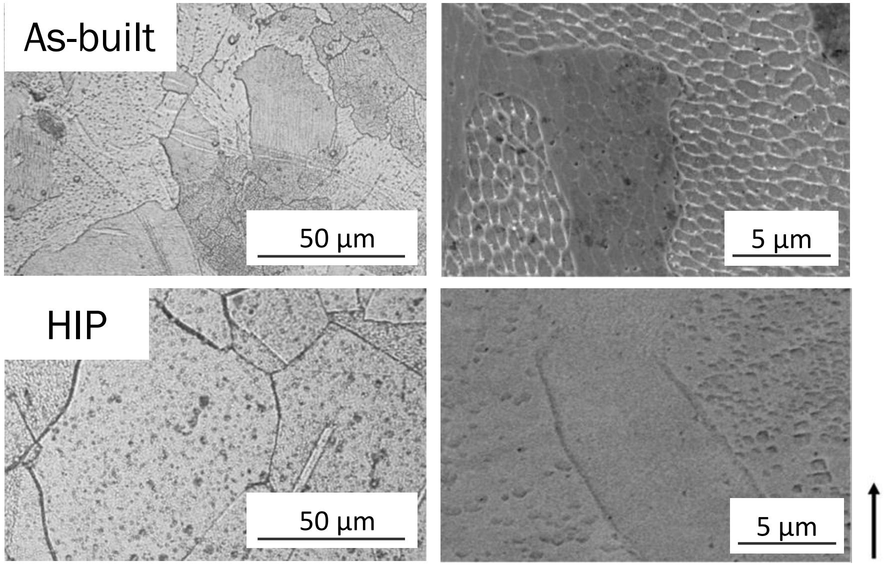


HIP



Giovagnoli, Maverick, et al. "Effect of different heat-treatment routes on the impact properties of an additively manufactured AlSi10Mg alloy." *Materials Science and Engineering: A* (2020): 140671.

HIP: microstructural effect



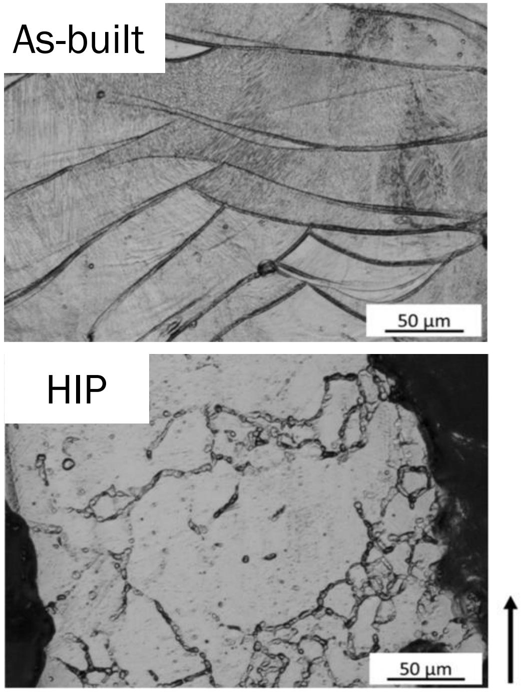
316L

Prior to HIP: cellular microstructure
Post HIP: large equiaxed grains.

CoCrMo

Prior to HIP: cellular microstructure
Post HIP: large equiaxed grains.

Melt pool boundaries are no longer visible after HIP.



Montero-Sistiaga, Maria L., et al. "Microstructure evolution of 316L produced by HP-SLM (high power selective laser melting)." *Additive Manufacturing* 23 (2018): 402-410.

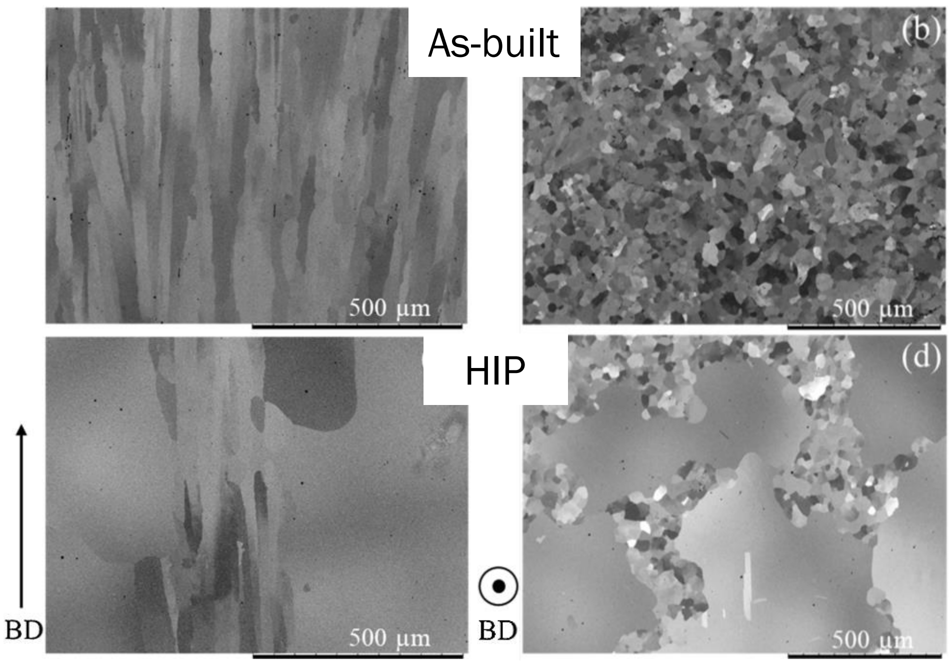
A. Cutolo, et al. "Influence of layer thickness and post-process treatments on the fatigue properties of CoCr scaffolds produced by laser powder bed fusion", *Addit. Manuf.* 23 (2018) 498–504.

HIP: microstructural effect

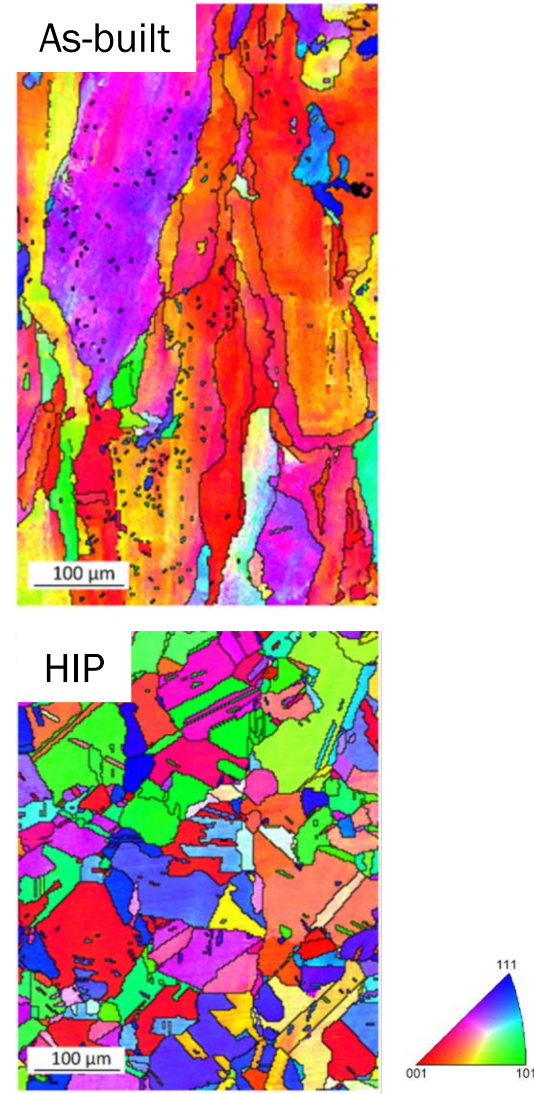
Ni-based alloys:

IN718: abnormal grain growth
IN625: recrystallization, texture randomization

IN718



IN625



Goel, Sneha, et al. "Effect of post-treatments under hot isostatic pressure on microstructural characteristics of EBM-built Alloy 718." *Additive Manufacturing* 28 (2019): 727-737.
Kreitcberg, Alena, et al. "Effect of heat treatment and hot isostatic pressing on the microstructure and mechanical properties of Inconel 625 alloy processed by laser powder bed fusion." *Materials Science and Engineering: A* 689 (2017): 1-10.

HIP: microstructural effect (global)

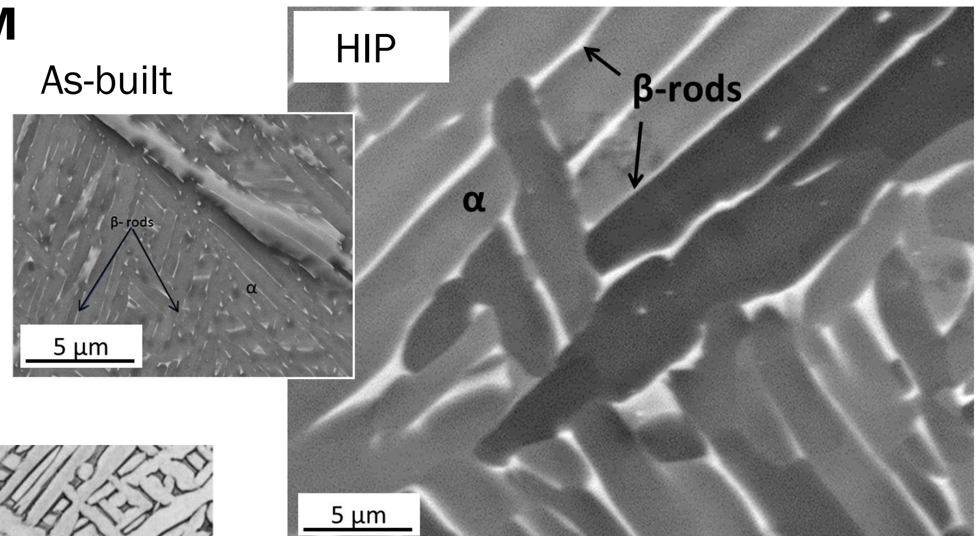
Ti-6Al-4V

EBM/DED: microstructural coarsening

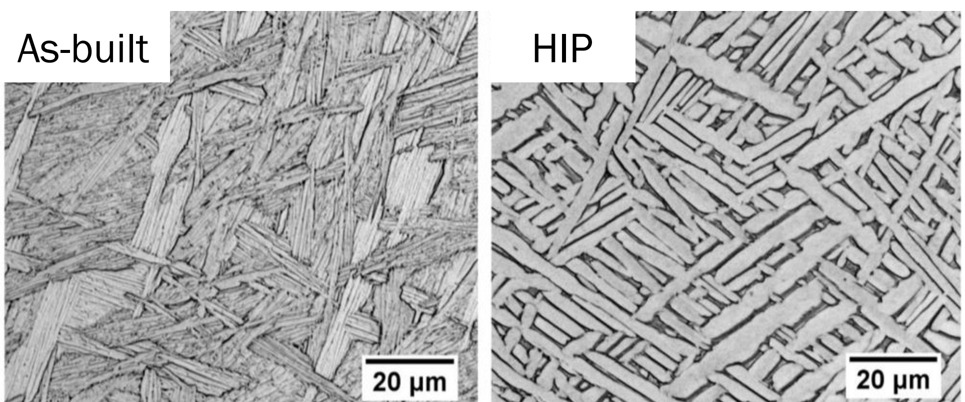
α lath thickness increases from 1-2 µm in as-built condition to 3-5 µm after HIP

EBM

As-built



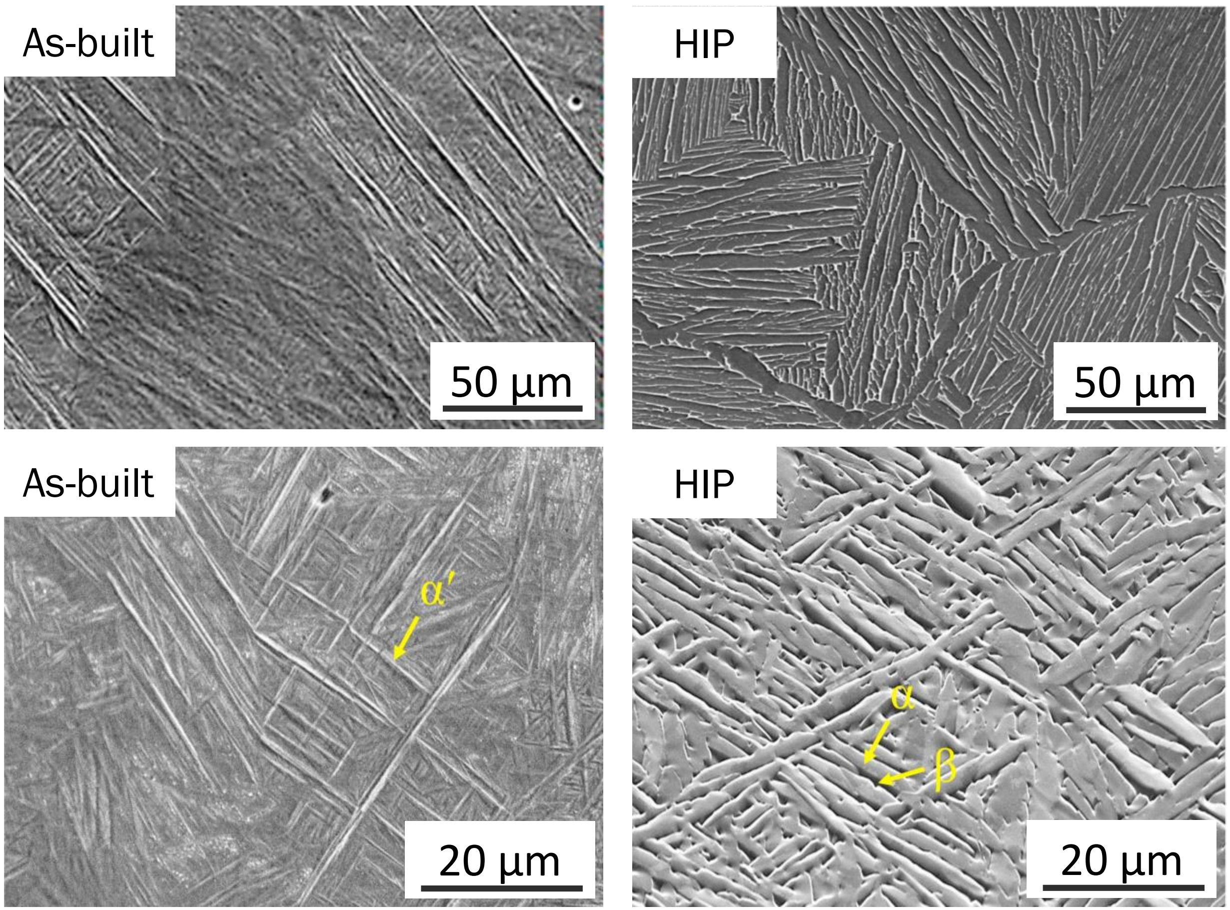
DED



HIP: microstructural effect (global)

Ti-6Al-4V

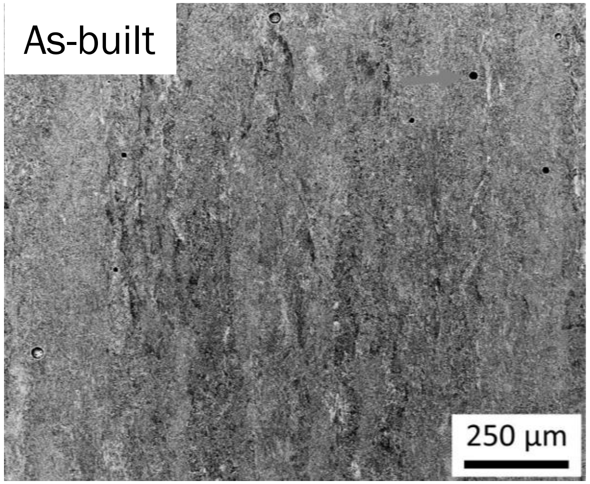
LPBF: martensite decomposition α’ ⟶ α+β and microstructural coarsening
α lath thickness of about 4 μm after HIP

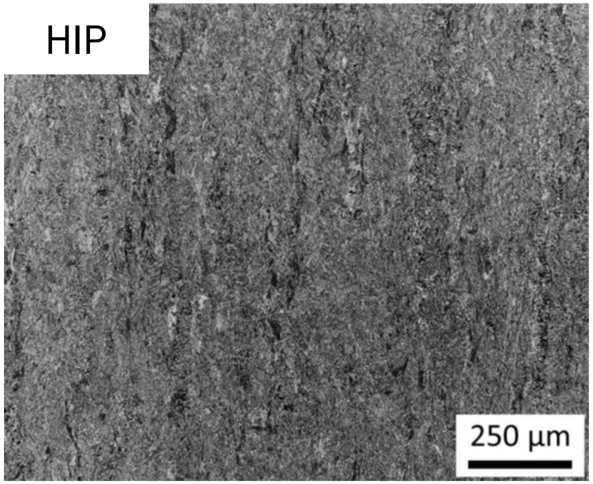


HIP: microstructural effect (global)

Ti-6Al-4V

HIP has no effect on parent β grains columnar morphology (because HIP is performed below the transus temperature).

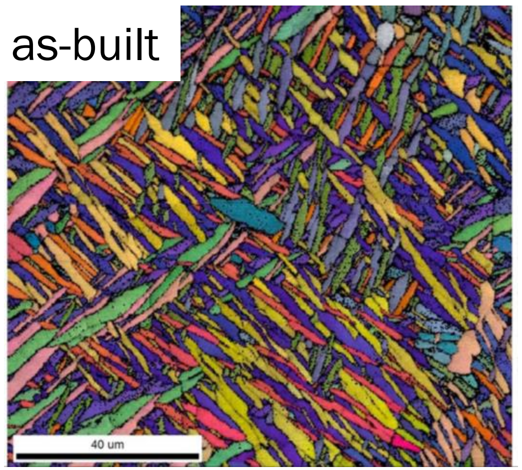


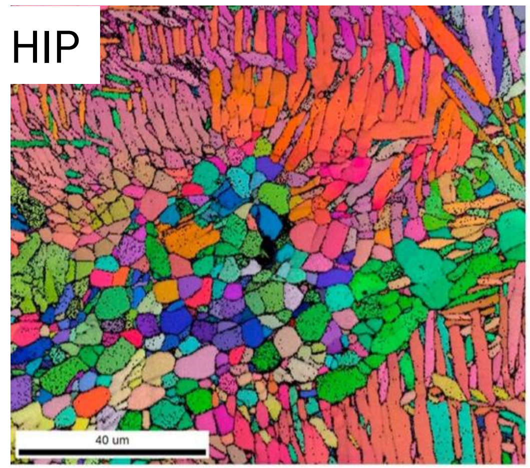


HIP: microstructural effects (in the vicinity of pores)

Ti-6Al-4V

A fine equiaxed microstructure is observed at the locations of former defects.





High-temperature deformation takes place **near voids** during HIP:
local hot working, with substantial deformation in the $\alpha+\beta$ temperature range.
Other locations of the specimen undergo less deformation during compaction.
$\Rightarrow$ more refined post-HIP microstructure near defects

Li, P., et al. "Investigation of the mechanisms by which hot isostatic pressing improves the fatigue performance of powder bed fused Ti-6Al-4V." *International journal of fatigue* 120 (2019): 342-352.

Post-treatments for microstructure and properties control

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Stress-relief: process conditions

Stress relief SR: thermal post-treatment allowing to reduce or eliminate residual stresses.

Objective: decrease distortions before cutting the parts from the building platform, without significantly affecting the grain structure.

1. uniform heating to a suitable temperature below the transformation range,
2. holding at this temperature for a given period of time,
3. uniform cooling

Material	**Temperature**
Co-Cr-Mo	450°C/45min + 750°C/1h/furnace cooling FC
Ni-based alloys (IN625, IN718)	650-870°C/1-8h
Steels (316L, AISI 4340)	470-650°C/40min-5h
Ti-6Al-4V	600-750°C/2-3h/furnace cooling or air cooling
Al alloys (4xxx serie: Al-Si)	150-400°C/2-6h/furnace cooling or air cooling

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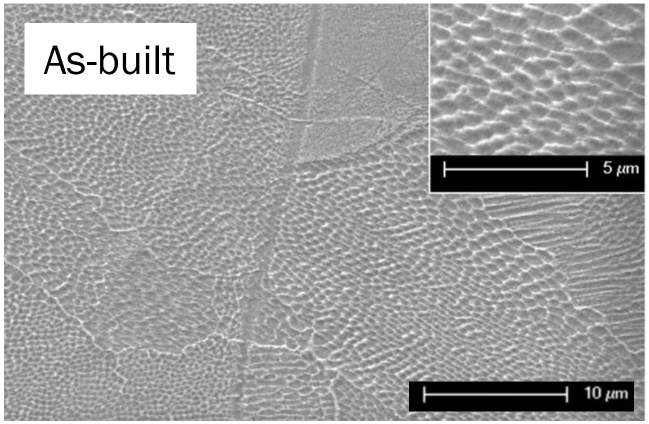
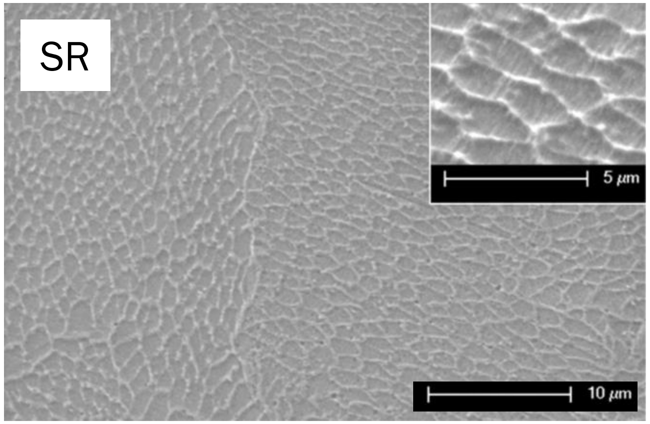
Stress-relief: microstructural effect

In some cases (e.g. steels), the SR temperature is too low for major microstructural changes to occur.

ex: **316L** – SR at 470°C – slight coarsening of the cellular solidification morphology





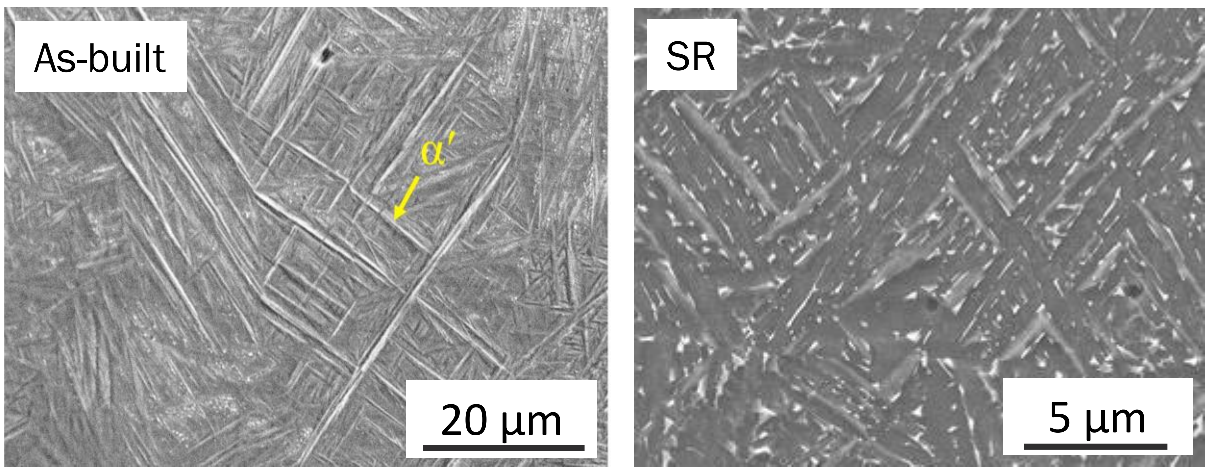
Condition	$\sigma_{y,0.2\%}$[MPa]	UTS [MPa]	Elongation before fracture [%]
As-built	453 ± 6	573 ± 6	46 ± 1
Stress relief heat treated	449 ± 5	570 ± 5	48 ± 1

Elangeswaran, Chola, et al. "Effect of post-treatments on the fatigue behaviour of 316L stainless steel manufactured by laser powder bed fusion." *International Journal of Fatigue* 123 (2019): 31-39.

Stress-relief: microstructural effect

In other cases (e.g. Al alloys, Ti64) the SR treatment can have a more significant effect

<u>ex</u>: **Ti64** – SR at 730°C/2h – decomposition of the martensite α' → α+β



Vilaro, Thomas, Christophe Colin, and Jean-Dominique Bartout. "As-fabricated and heat-treated microstructures of the Ti-6Al-4V alloy processed by selective laser melting." *Metallurgical and materials transactions A* 42.10 (2011): 3190-3199.

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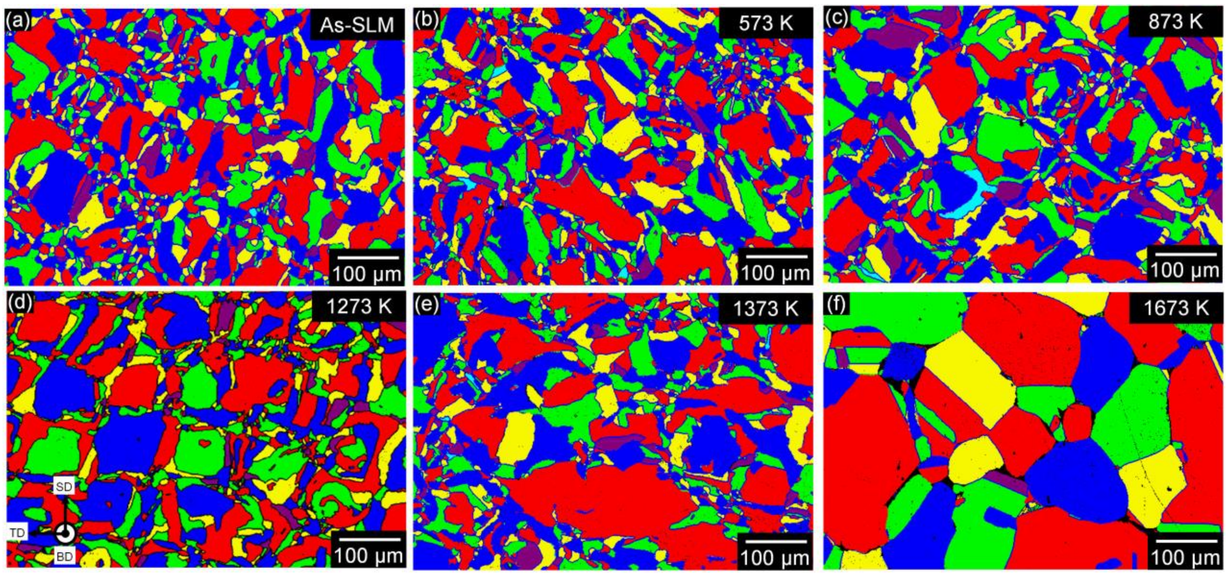
Heat treatments: typical microstructural effects

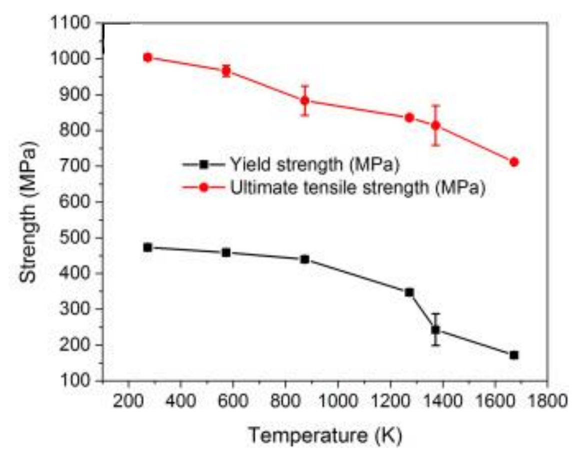
Grain growth

<u>ex</u>: **316L**



Average grain size
as-built: 45 ± 1 µm
300 °C: 50 ± 3 µm
600 °C: 55 ± 3 µm
1000 °C: 65 ± 8 µm
1100 °C: 88 ± 5 µm
1400 °C: 102 ± 3 µm

The grains become more equiaxed and larger with increasing annealing temperature.



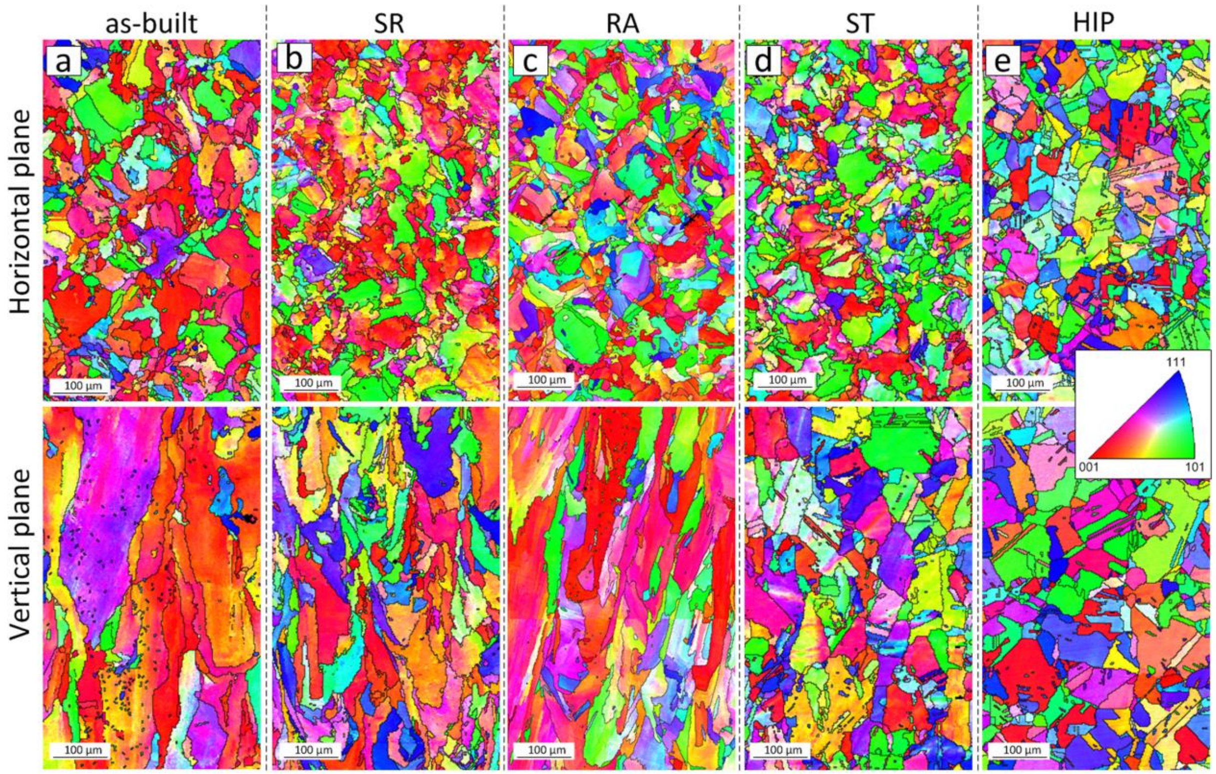
Salman, O. O., et al. "Effect of heat treatment on microstructure and mechanical properties of 316L steel synthesized by selective laser melting." *Materials Science and Engineering: A* 748 (2019): 205-212.

Heat treatments: typical microstructural effects

Recrystallization

ex: **IN625**

SR: 650-870°C
RA: 930-1040°C
ST: 1040-1200°C
HIP: 1120–1240°C



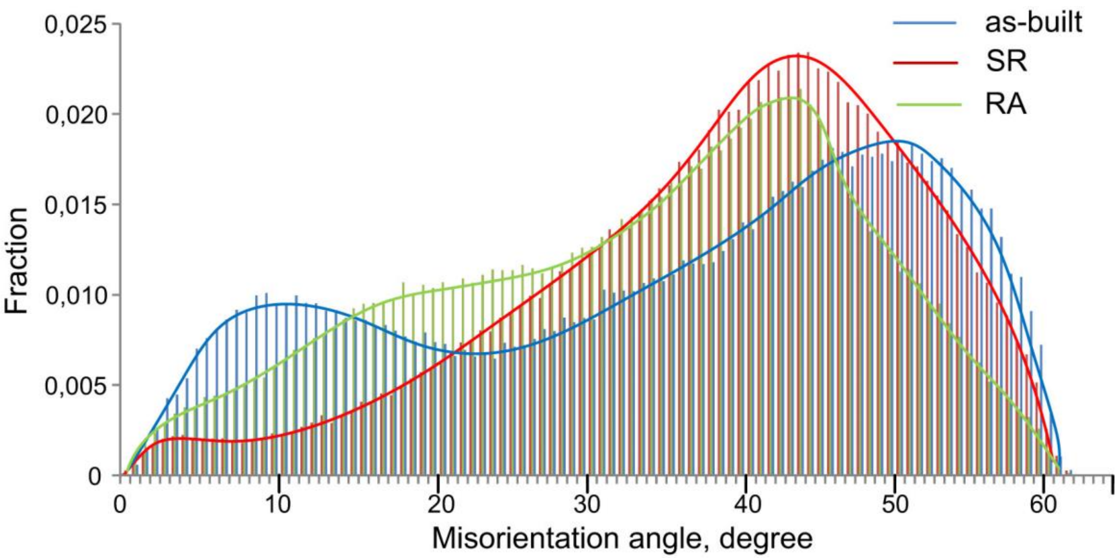
Kreitcberg, Alena, et al. "Effect of heat treatment and hot isostatic pressing on the microstructure and mechanical properties of Inconel 625 alloy processed by laser powder bed fusion." *Materials Science and Engineering: A* 689 (2017): 1-10.

Heat treatments: typical microstructural effects

Recrystallization

ex: **IN625**

As-built:
bimodal misorientation angle distribution
⇒ mixed grain/subgrain structure



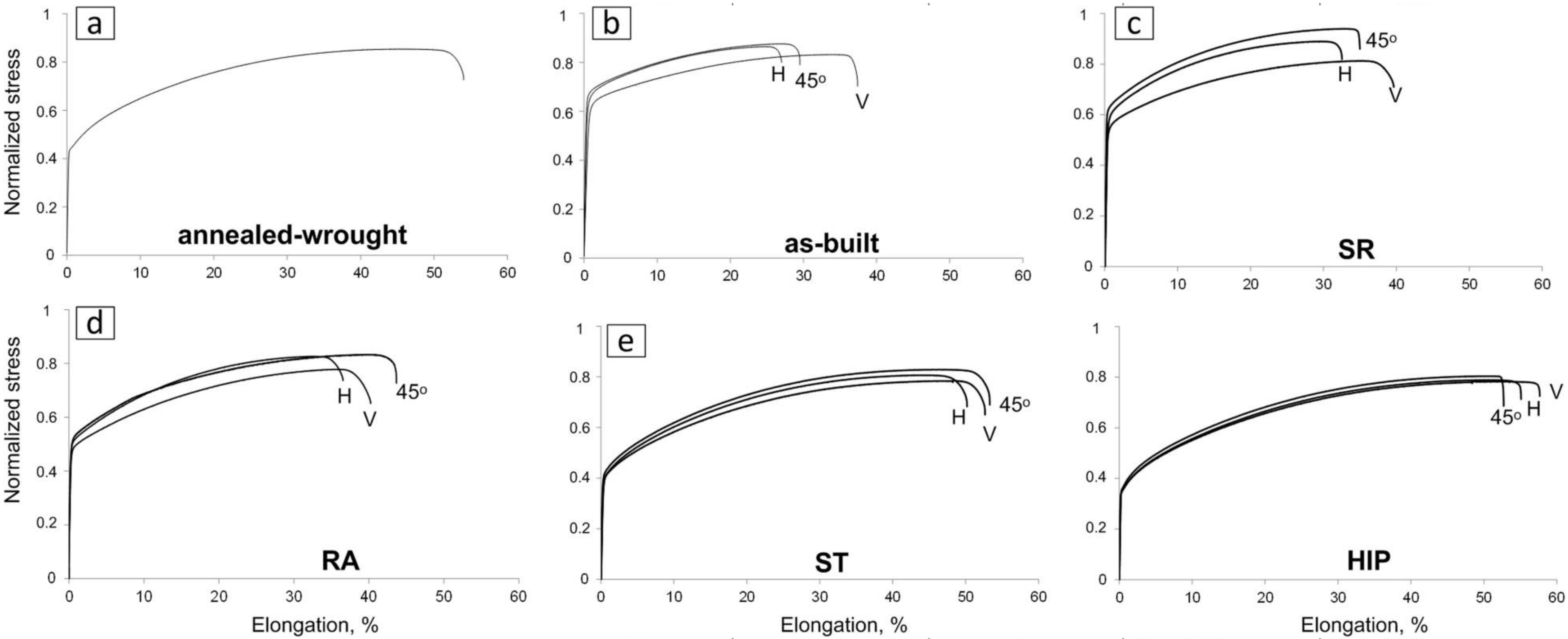
SR:
The fraction of low-angle grain boundaries decreases, while the fraction of high-angle grain boundaries increases
⇒ coalescence of subgrains

RA:
The amount of grain boundaries with 15–30° misorientation increases
⇒ partial recrystallization

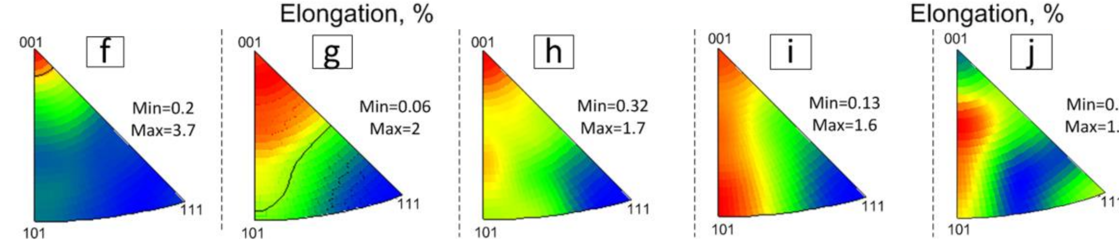
Kreitcberg, Alena, et al. "Effect of heat treatment and hot isostatic pressing on the microstructure and mechanical properties of Inconel 625 alloy processed by laser powder bed fusion." *Materials Science and Engineering: A* 689 (2017): 1-10.

Heat treatments: typical microstructural effects

Recrystallization



Texture randomization
and reduced anisotropy



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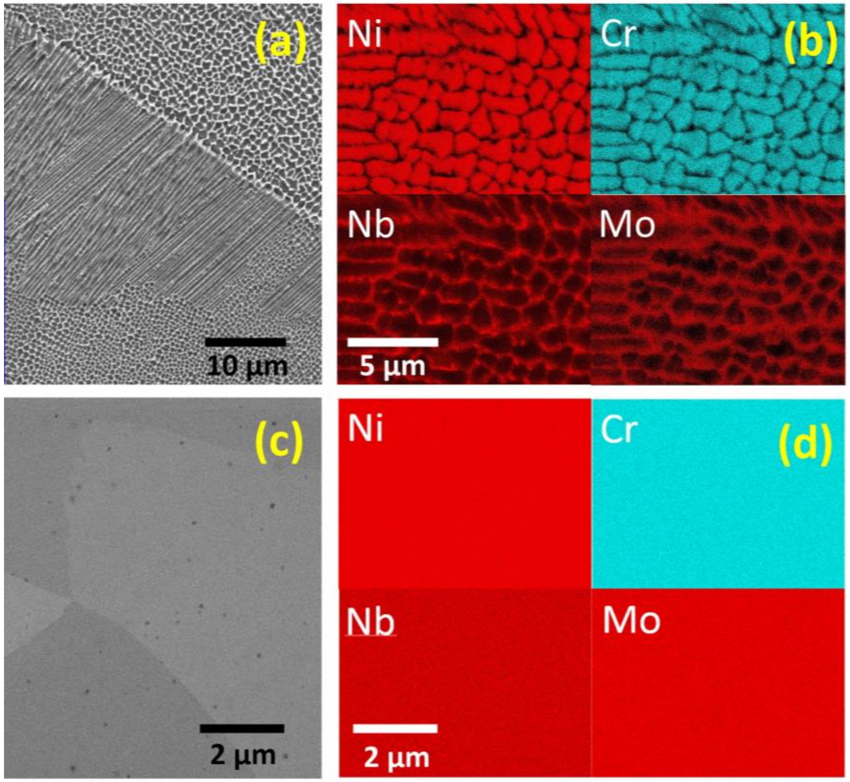
Heat treatments: typical microstructural effects

Microstructure homogenization

<u>ex</u>: **IN625**



As-built:
fine dendritic microstructures and elemental segregation due to rapid solidification

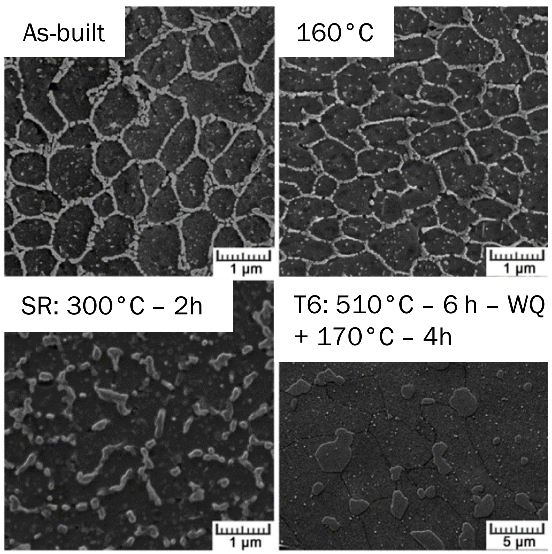
ST - 1150 °C - 1 h:
uniform chemical composition

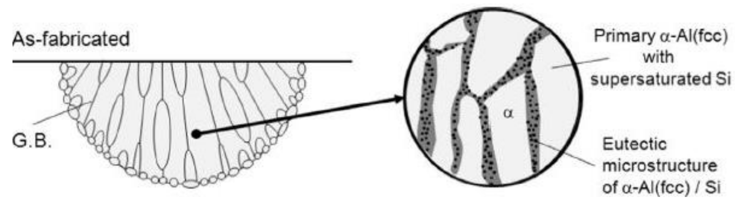
Zhang, Fan, et al. "Homogenization kinetics of a nickel-based superalloy produced by powder bed fusion laser sintering." *Scripta materialia* 131 (2017): 98-102.

Heat treatments: typical microstructural effects

Precipitation

ex: Al-Si alloys (4xxx series)





As-built cellular microstructure: primary α-Al columnar grains with a Si-rich network at the cell boundaries.

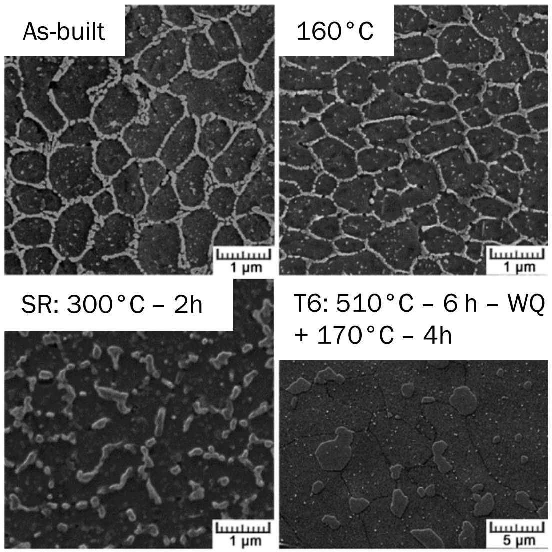
At annealing temperatures **as low as 160°C**: Precipitation of nano-sized pure Si in the matrix but unchanged intercellular Si-rich network.

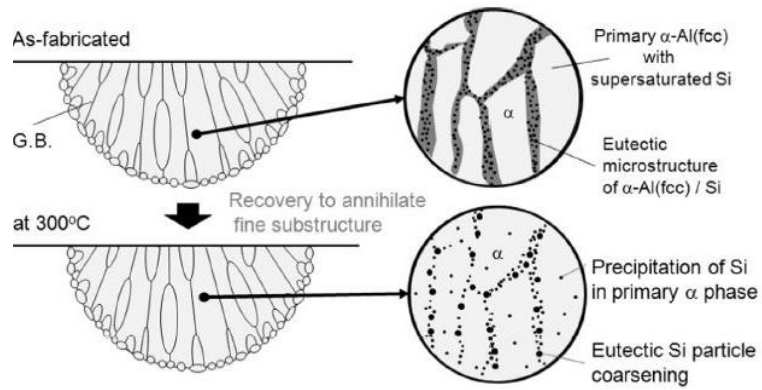
Fousová, Michaela, et al. "Changes in the microstructure and mechanical properties of additively manufactured AlSi10Mg alloy after exposure to elevated temperatures." *Materials Characterization* 137 (2018): 119-126.

Heat treatments: typical microstructural effects

Precipitation

<u>ex</u>: Al-Si alloys (4xxx series)





At 300°C: breaking up of the Si network acting as nucleation sites for the formation of Si particles

Diffusion of excess Si from the α-Al matrix

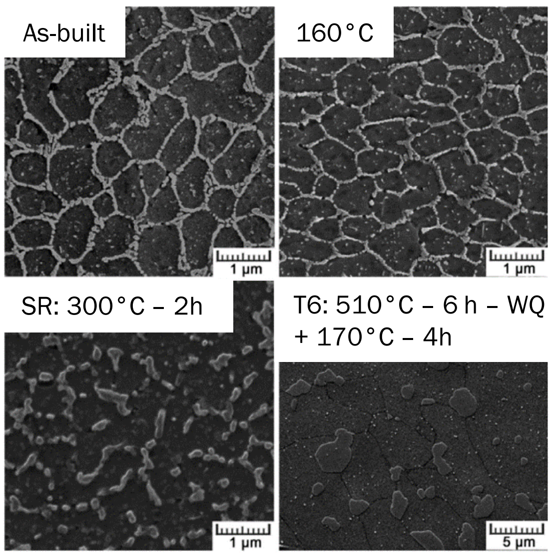
⇒ coarsening of the Si particles at these nucleation sites

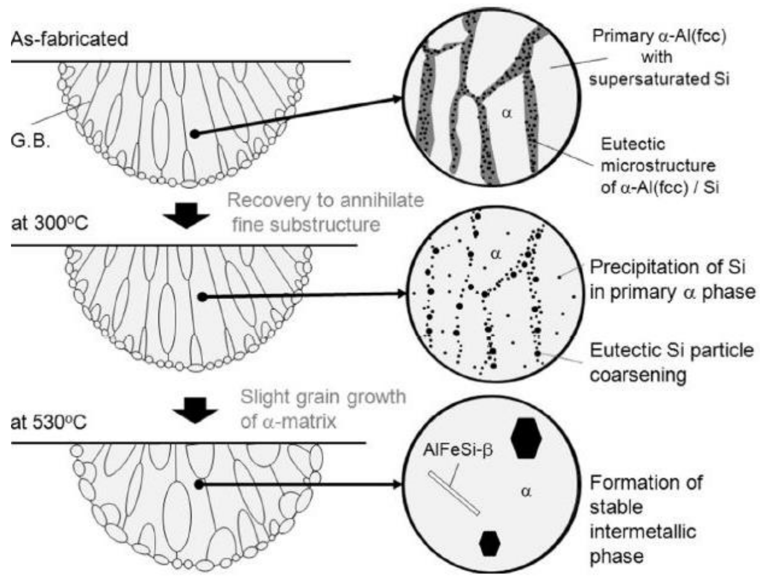
Fousová, Michaela, et al. "Changes in the microstructure and mechanical properties of additively manufactured AlSi10Mg alloy after exposure to elevated temperatures." *Materials Characterization* 137 (2018): 119-126.

Heat treatments: typical microstructural effects

Precipitation

ex: Al-Si alloys (4xxx series)





Above **500°C**: further grain growth, Si phase coarsening and formation of a stable iron intermetallic phase

Fousová, Michaela, et al. "Changes in the microstructure and mechanical properties of additively manufactured AlSi10Mg alloy after exposure to elevated temperatures." *Materials Characterization* 137 (2018): 119-126.

Heat treatments: typical microstructural effects

Precipitation

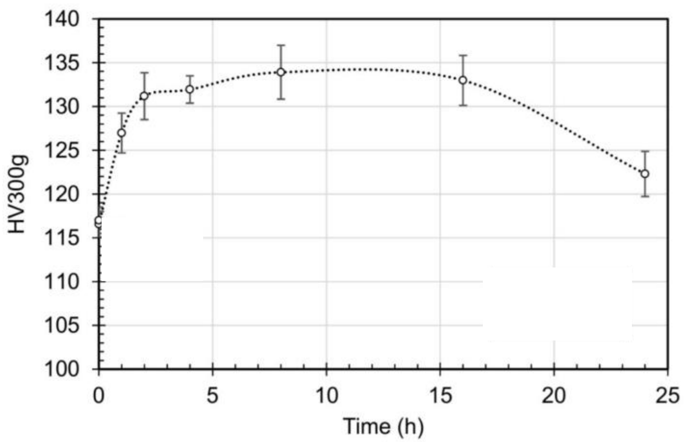
ex: Al-Si alloys (4xxx series) – direct aging T5

Skip the solution-annealing stage (first step of the T6 treatment) which induces grain growth and coarsening of the Si particles

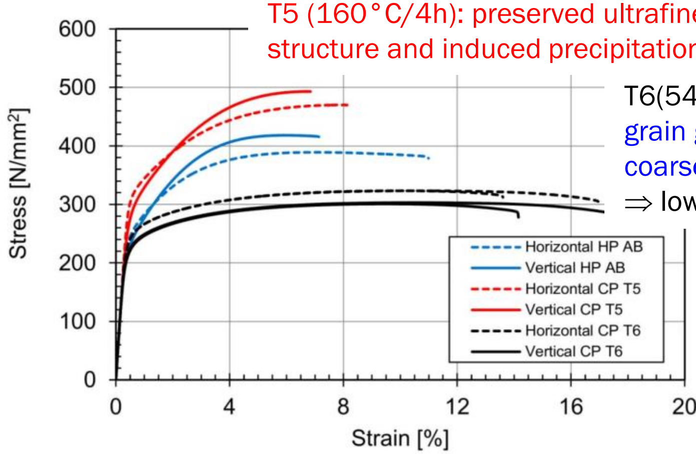
⇒ Promote precipitation aging while starting right from the as-built material

⇒ Exploit

- supersaturation of the solid solution to enhance precipitation strengthening
- fine cellular structure to maximize strengthening



Vickers' microhardness evolution as a function of holding time at the aging temperature of 160°C



T5 (160°C/4h): preserved ultrafine cellular structure and induced precipitation strengthening

T6(540°C/1h/WQ+160°C/4h): grain growth and Si particles coarsening

⇒ low strength, high ductility

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  - typical microstructural effects on other alloys
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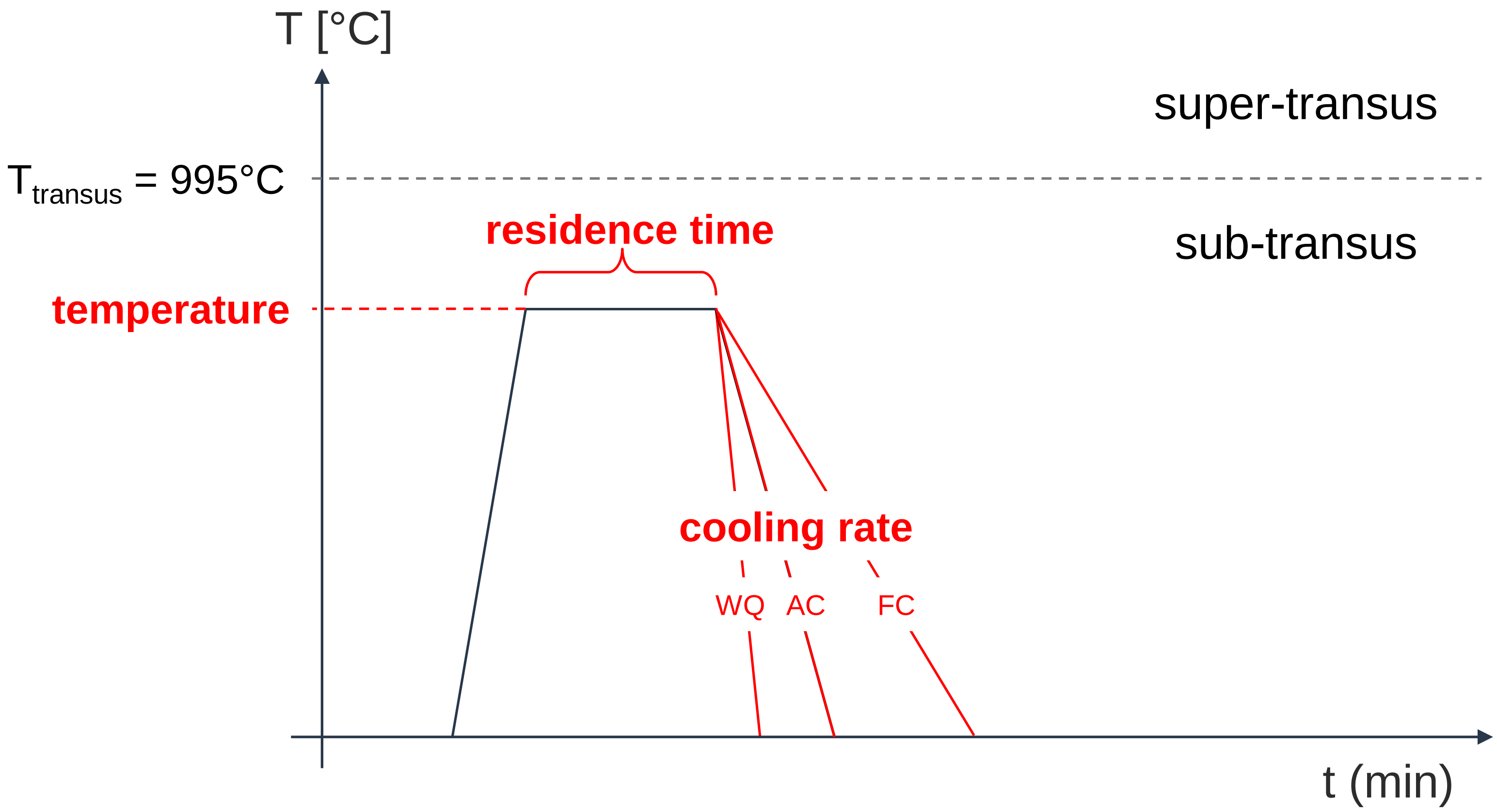
Thermal post-treatments of Ti-6Al-4V

Effect of heat treatments: influencing factors

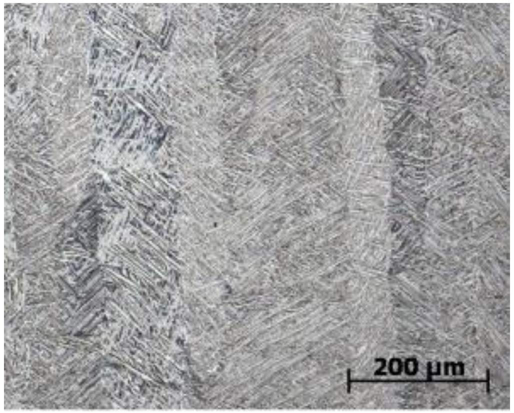


WQ : water quenching
AC : air cooling
FC : furnace cooling

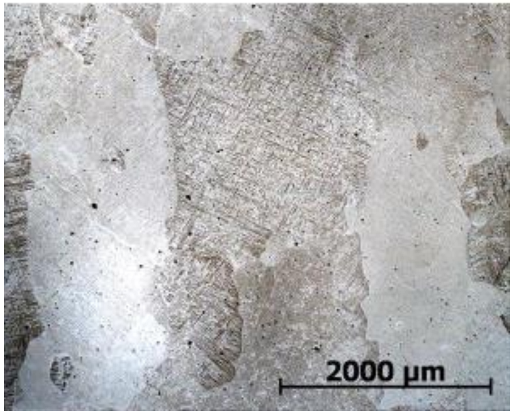
Thermal post-treatments of Ti-6Al-4V

Effect of heat treatments: influencing factors

Temperature



Sub-transus temperature (940 °C):
the columnar microstructure is maintained, the growth of the β grain is hindered by the presence of α phase along the β grain boundaries



Super-transus temperature (1015 °C):
uncontrolable β grain growth (several mm)

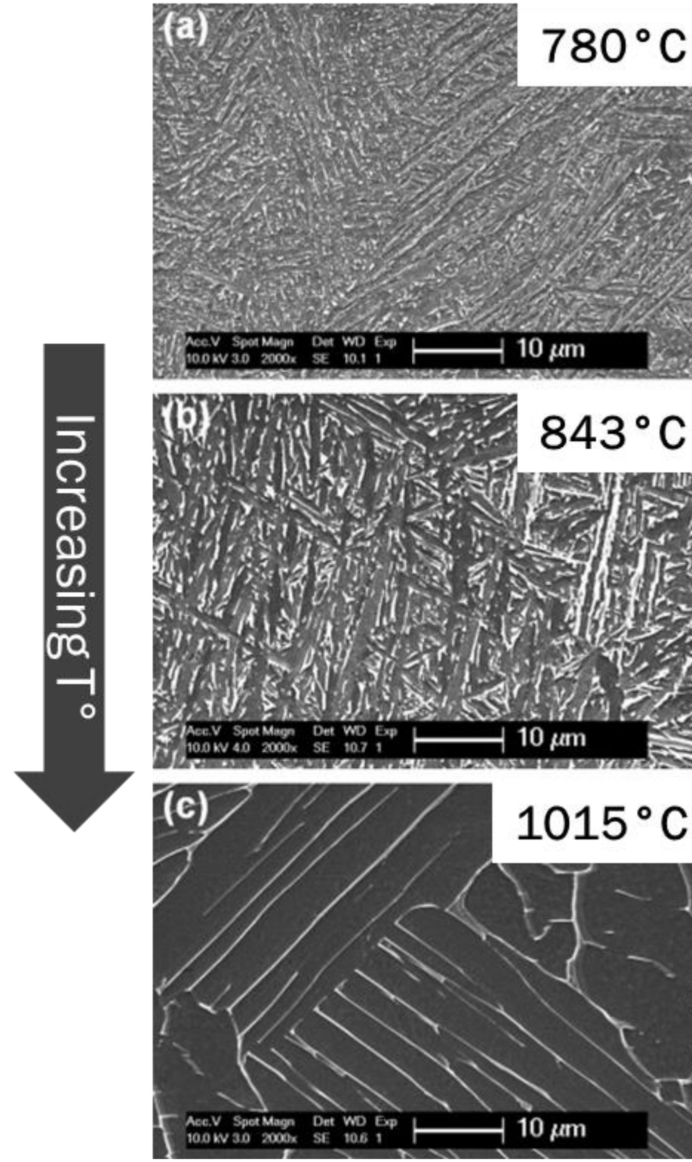
B. Vrancken et al., *Journal of Alloys and Compounds*, 2012

Thermal post-treatments of Ti-6Al-4V

Effect of heat treatments: influencing factors

Temperature

At high temperatures, martensite decomposition α’ → α + β and microstructural coarsening result in coarse α lamellae delinated by β phase.



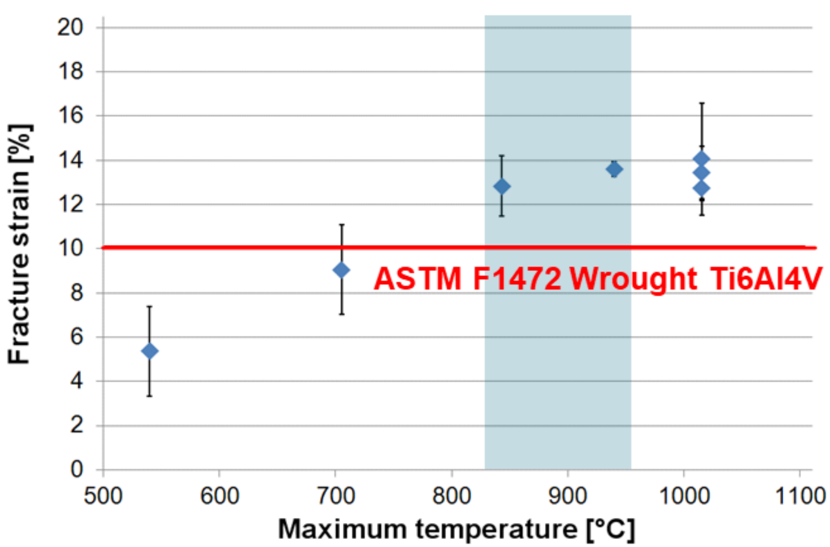
B. Vrancken et al., *Journal of Alloys and Compounds*, 2012

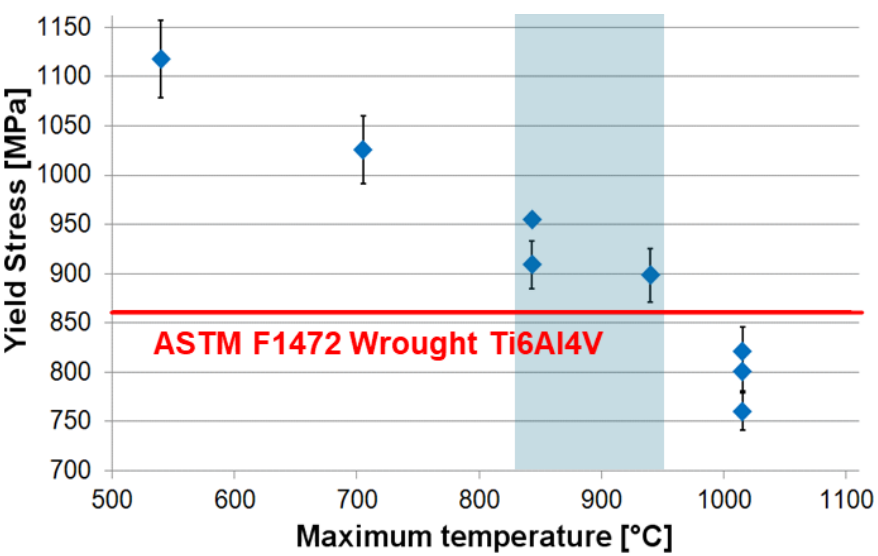
Thermal post-treatments of Ti-6Al-4V

Effect of heat treatments: influencing factors

Temperature

Martensite decomposition substantially increases the ductility of the material but reduces the strength. For LPBF Ti6Al4V, the optimal heat treatment temperature ranges between 850 °C and 950 °C.



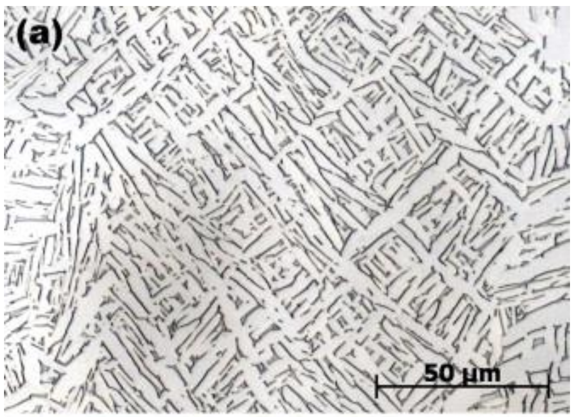


B. Vrancken et al., *Journal of Alloys and Compounds*, 2012

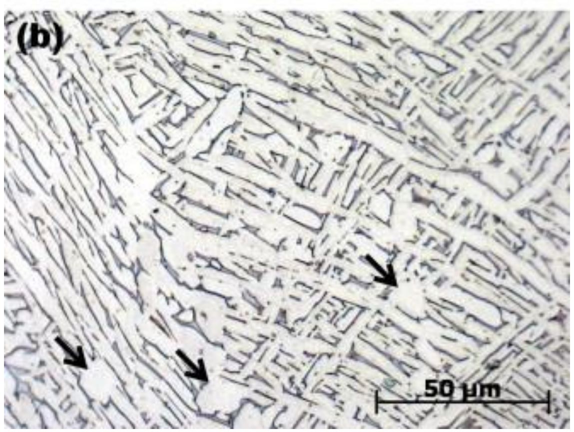
Thermal post-treatments of Ti-6Al-4V

Effect of heat treatments: influencing factors

Residence time



2h at 940°C



20h at 940°C

At sub-transus temperatures, the residence time has a very limited influence on the microstructure.

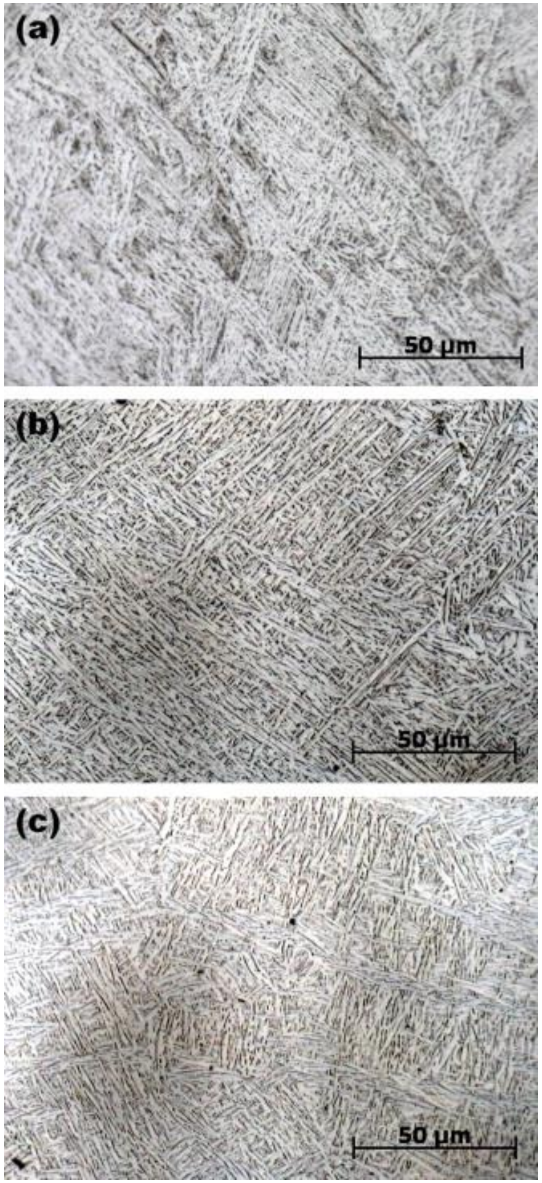
B. Vrancken et al., *Journal of Alloys and Compounds*, 2012

Thermal post-treatments of Ti-6Al-4V

2h at 850°C

Effect of heat treatments: influencing factors

Cooling rate



Furnace cooling

Air cooling

Water cooling

The influence of the cooling rate on the resulting microstructure increases as the annealing temperature increases.

At low subtransus temperatures (<850°C), the cooling rate has a minor effect on the microstructure (similar α needle sizes).

B. Vrancken et al., *Journal of Alloys and Compounds*, 2012

Thermal post-treatments of Ti-6Al-4V

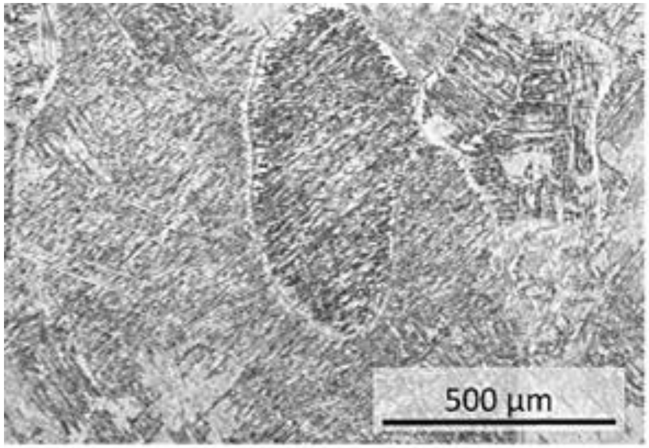
Effect of heat treatments: influencing factors

Cooling rate

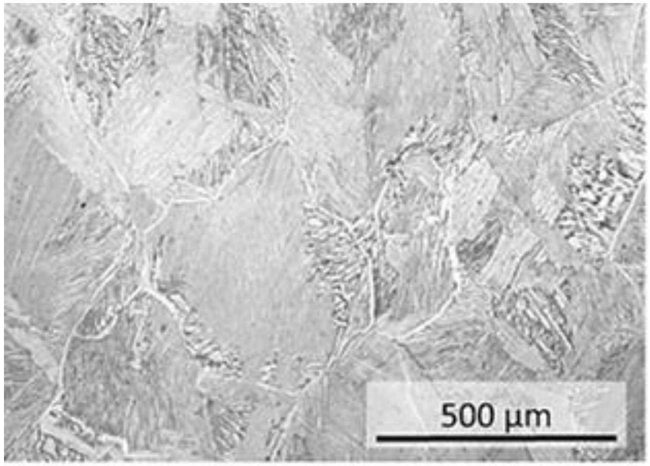
At temperatures above the transus, the cooling rate is the most important parameter. It determines the size and morphology of the α phase.

30 min at 1040°C

Air cooling
Very fine α lamellae



Furnace cooling
Large colonies of coarse α lamellae



B. Vrancken et al., *Journal of Alloys and Compounds*, 2012

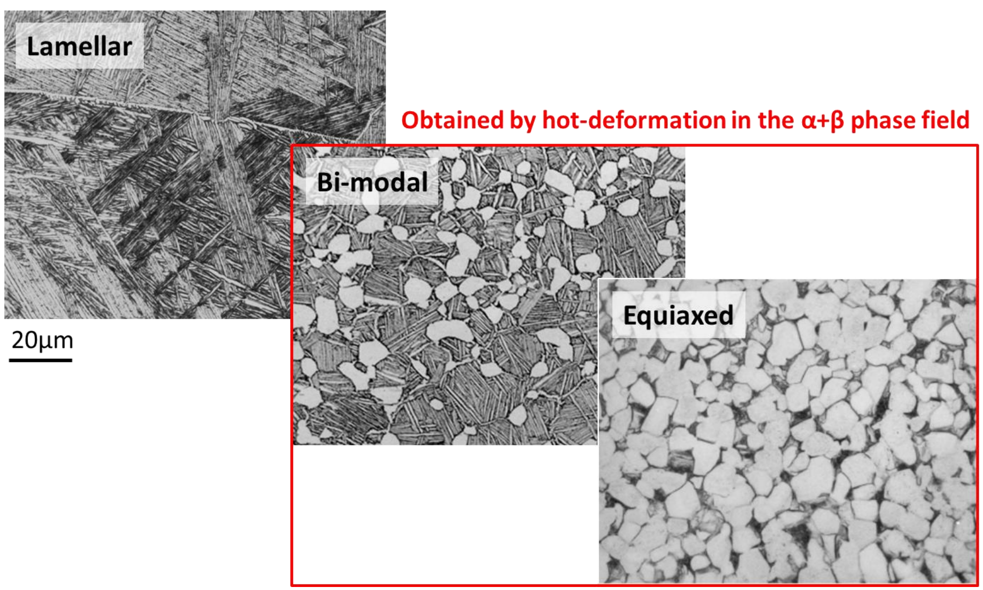
Thermal post-treatments of Ti-6Al-4V

SR heat treatments have a limited influence on the microstructure.
The following features are maintained after SR:

- columnar β grain morphology: **anisotropy**
- fine lamellar microstructure: high strength, **low ductility**

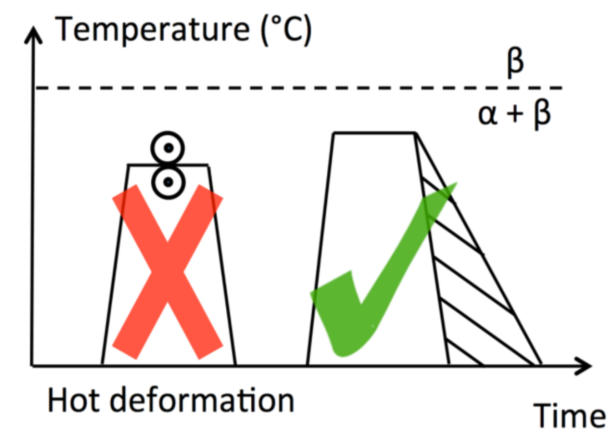
<u>Conventional</u> <u>metallurgy of Ti</u>

Hot deformation is used to obtain a microstructure with good mechanical properties (i.e. bi-modal or equiaxed).



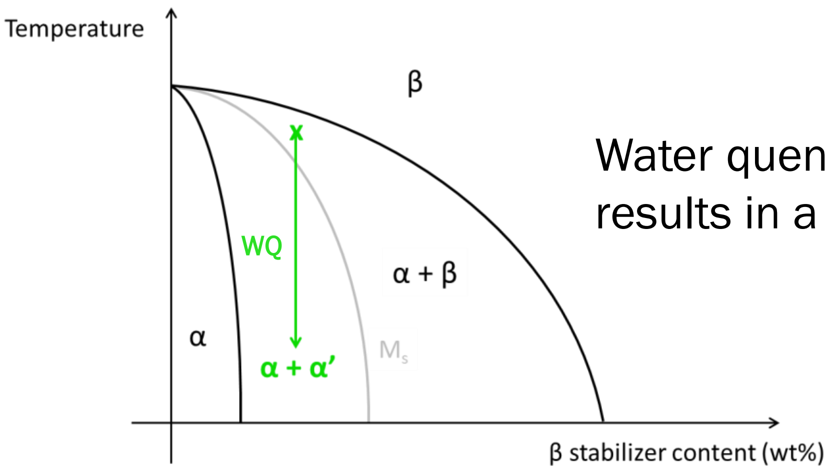
<u>Post-processing of</u> **<u>AM Ti</u>**

Major limitation for microstructural control: hot deformation cannot be performed on near-net shape AM parts.



Thermal post-treatments of Ti-6Al-4V

Engineering new heat treatments to tailor the microstructure of AM Ti64



Water quenching from the α+β domain (850 °C - 995 °C) results in a α+α' microstructure.

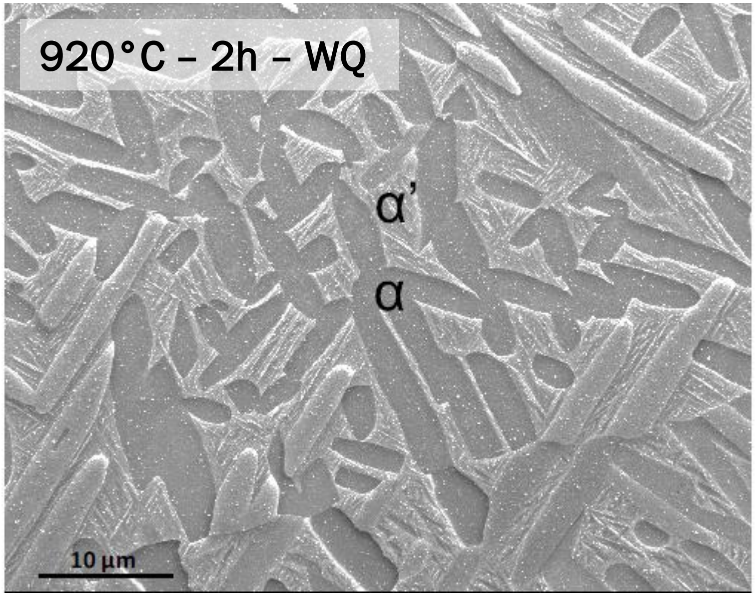
<u>Example</u>: 2h at 920 °C + water quenching WQ

At 920 °C: 50% β + 50% α

WQ

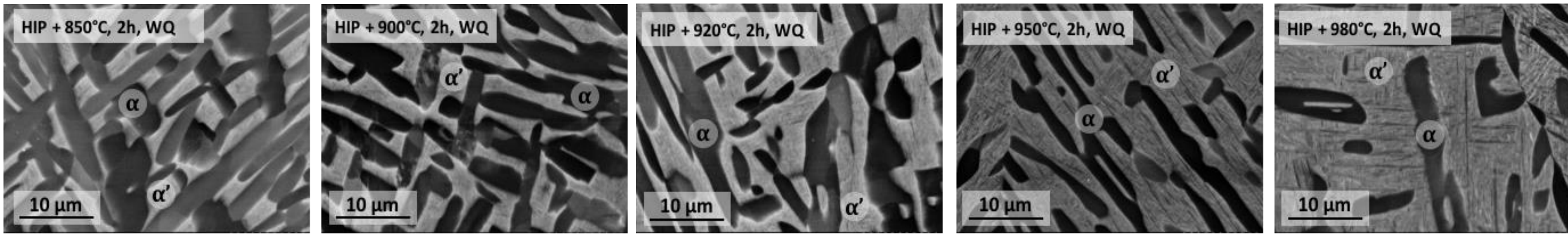
At 20 °C: 50% α' + 50% α

The "dual-phase" α+α' microstructure exhibits a combination of high strength and ductility.



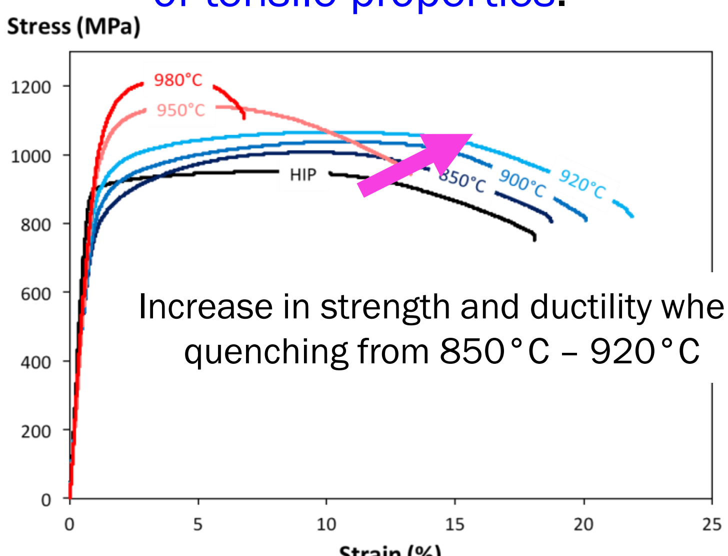
Thermal post-treatments of Ti-6Al-4V

Engineering new heat treatments to tailor the microstructure of AM Ti64

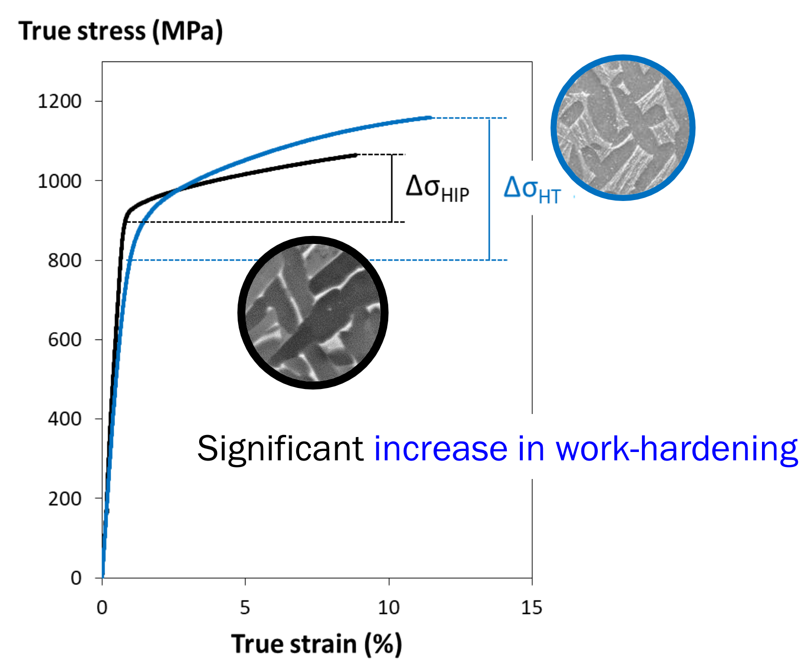


Depending on the temperature, various fractions of α' martensite are obtained.

This results in a broad range of tensile properties.



Increase in strength and ductility when quenching from 850 °C – 920 °C

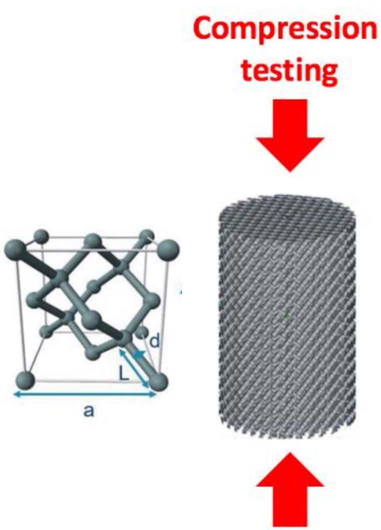


Significant increase in work-hardening

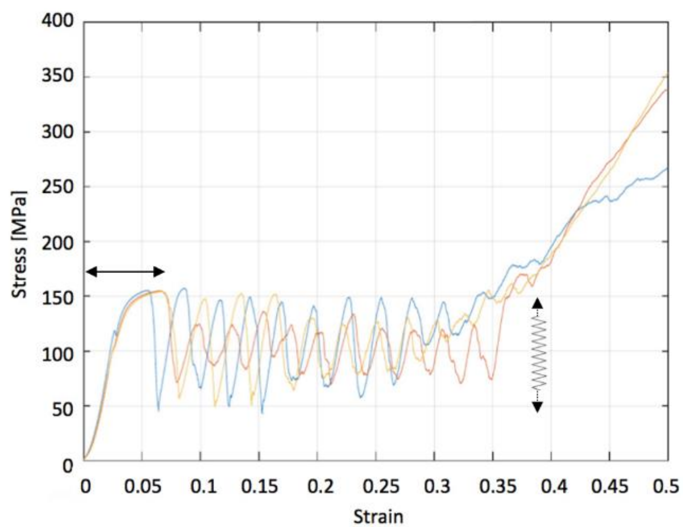
Thermal post-treatments of Ti-6Al-4V

Engineering new heat treatments to tailor the microstructure of AM Ti64

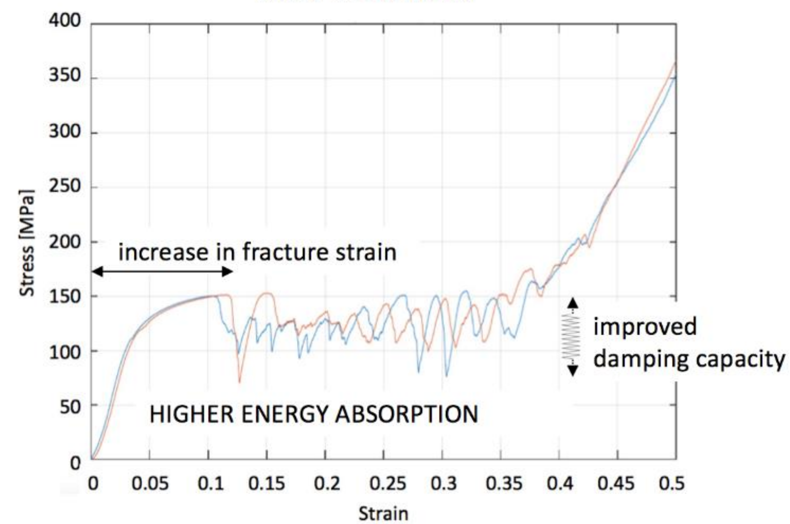
Ex. of application: energy absorption of porous structures



Stress-relief



Dual-phase heat treatment

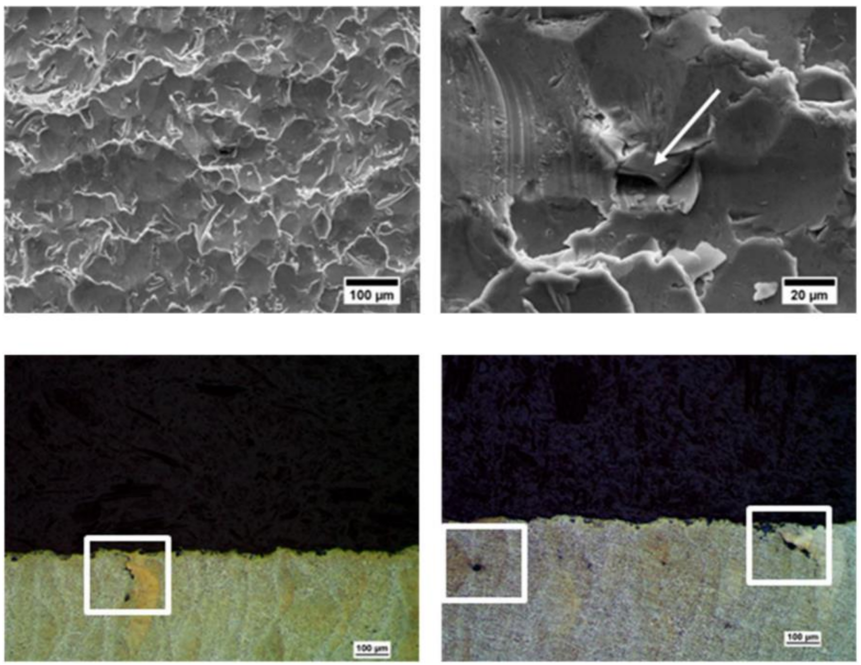


Post-treatments for microstructure and properties control

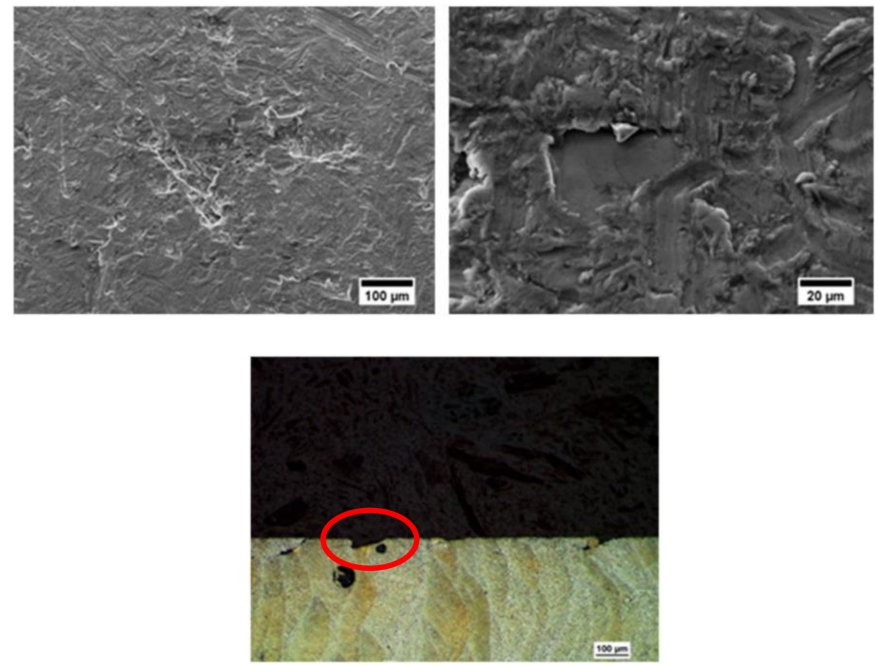
- HIP
  - process conditions
  - limitations
  - microstructural effects
- Stress-relief
  - process conditions
  - microstructural effects
- Thermal post-treatments for microstructure control
  - typical microstructural effects on other alloys
  - influencing factors (illustrated on Ti-6Al-4V)
- Surface post-treatments
- Effect of post-processing on mechanical properties
  - tensile
  - fatigue

Surface post-treatments

Sand blasting vs vibro-finishing



Sand blasting



Vibro-finishing

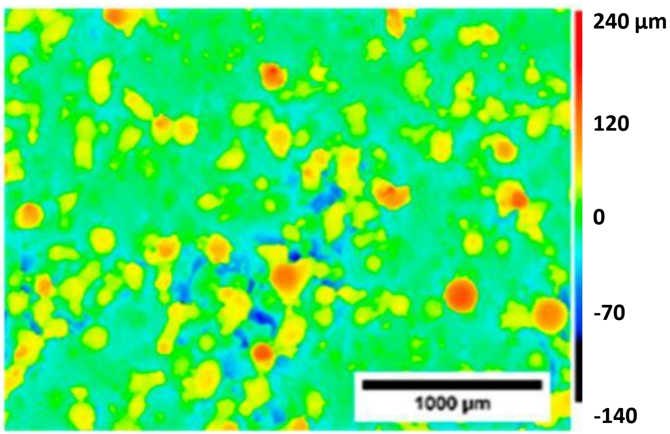
Open pores can still be identified on the surface profile.

⇒ inherited from surface or subsurface defects formed during L-PBF processing

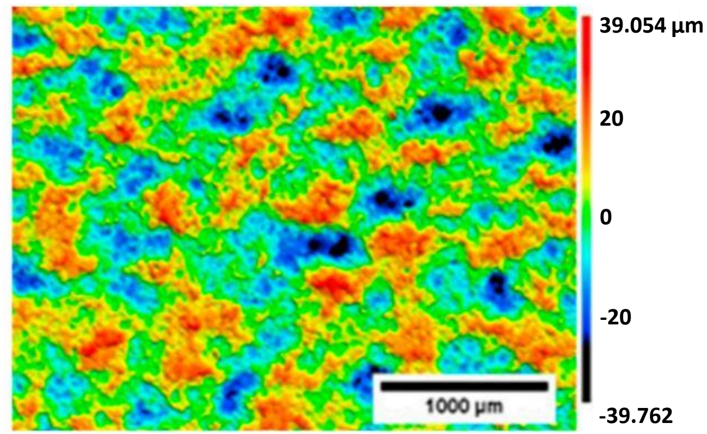
Hamidi Nasab, Milad, et al. "Effect of surface and subsurface defects on fatigue behavior of AlSi10Mg alloy processed by laser powder bed fusion (L-PBF)." *Metals* 9.10 (2019): 1063.

Surface post-treatments

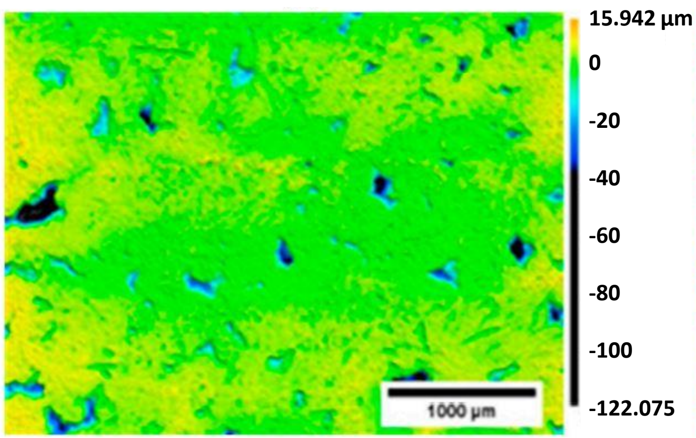
Sand blasting vs vibro-finishing



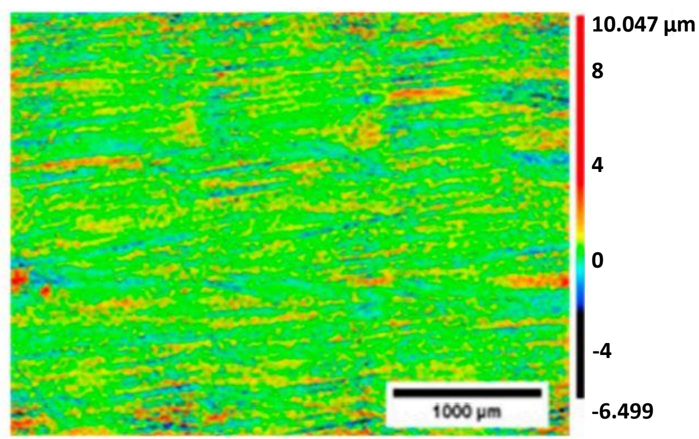
As-built



Sand-blasted



Vibro-finished



Machined and polished

Hamidi Nasab, Milad, et al. "Effect of surface and subsurface defects on fatigue behavior of AlSi10Mg alloy processed by laser powder bed fusion (L-PBF)." *Metals* 9.10 (2019): 1063.

Surface post-treatments

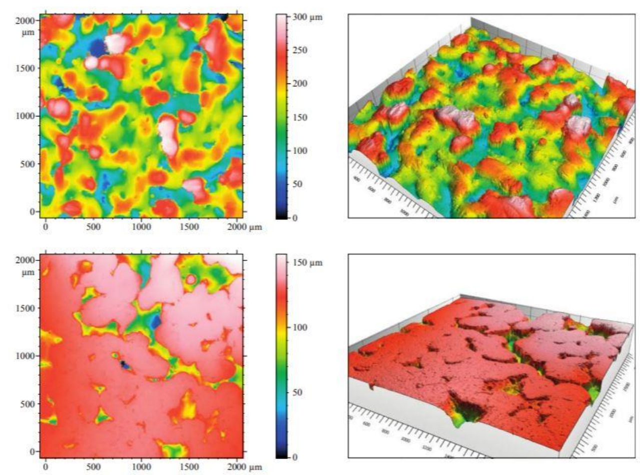
Sand blasting vs vibro-finishing

Surface Condition	S_a (μm)	S_v (μm)	S_{sk}
As-Built	15.4	85	0.74
SB	8.3	40	−0.13
VF	2.3	121	−7.31
MP	0.5	6	0.05

S_a arithmetical mean height
S_v maximum valley depth
S_{sk} skewness

Sand-blasting (SB) only partially reduces surface roughness, whereas vibro-finishing (VF) and machining+polishing (MP) are more effective in producing smooth surface

VF specimens contain deeper and more numerous valleys, as indicated by the negative skewness and higher S_v value

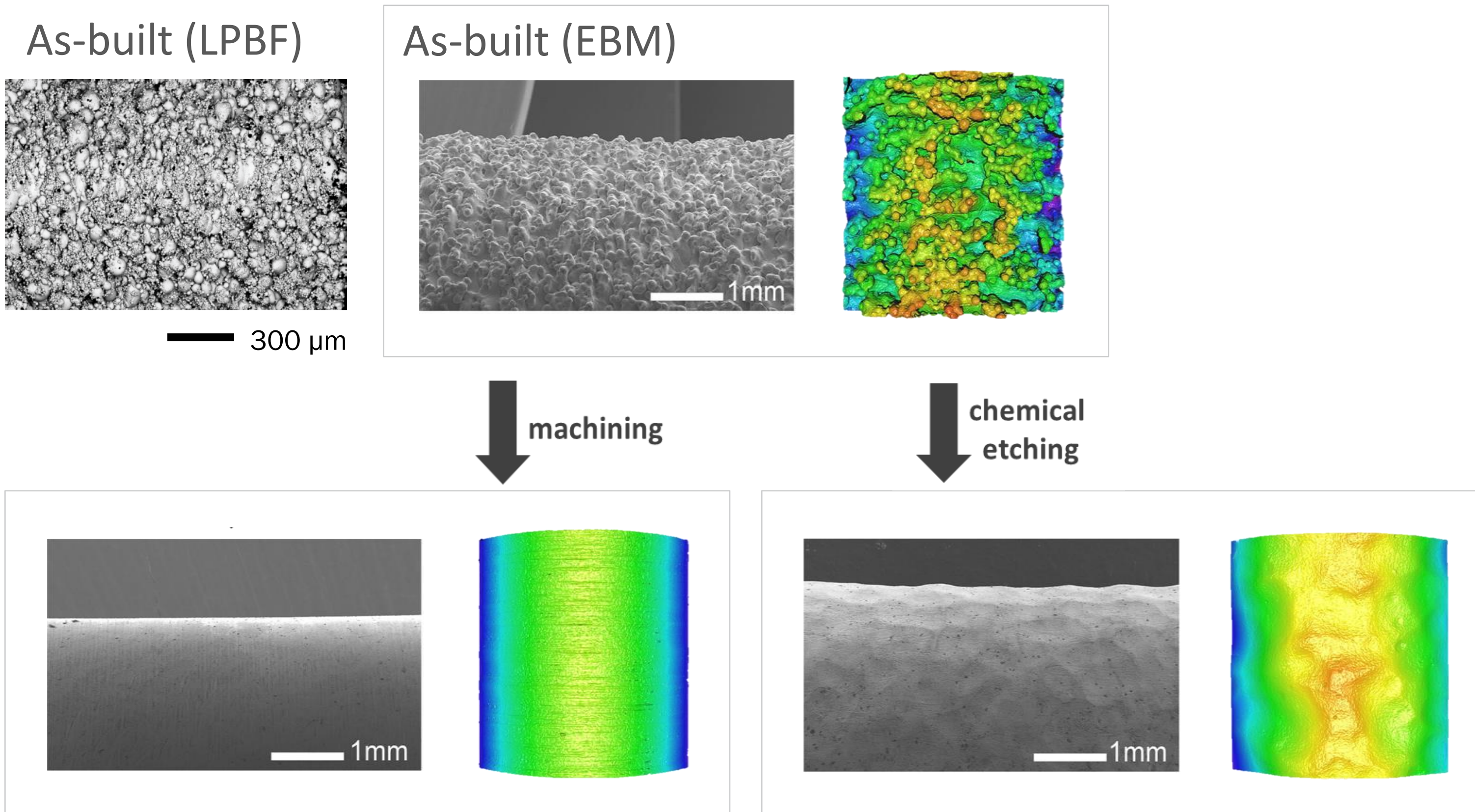


Hamidi Nasab, Milad, et al. "Effect of surface and subsurface defects on fatigue behavior of AlSi10Mg alloy processed by laser powder bed fusion (L-PBF)." *Metals* 9.10 (2019): 1063.
Atzeni, Eleonora, et al. "Performance assessment of a vibro-finishing technology for additively manufactured components." *Procedia CIRP* 88 (2020): 427-432.

Ti-6Al-4V

Surface post-treatments

Post-process treatment: machining vs chemical etching



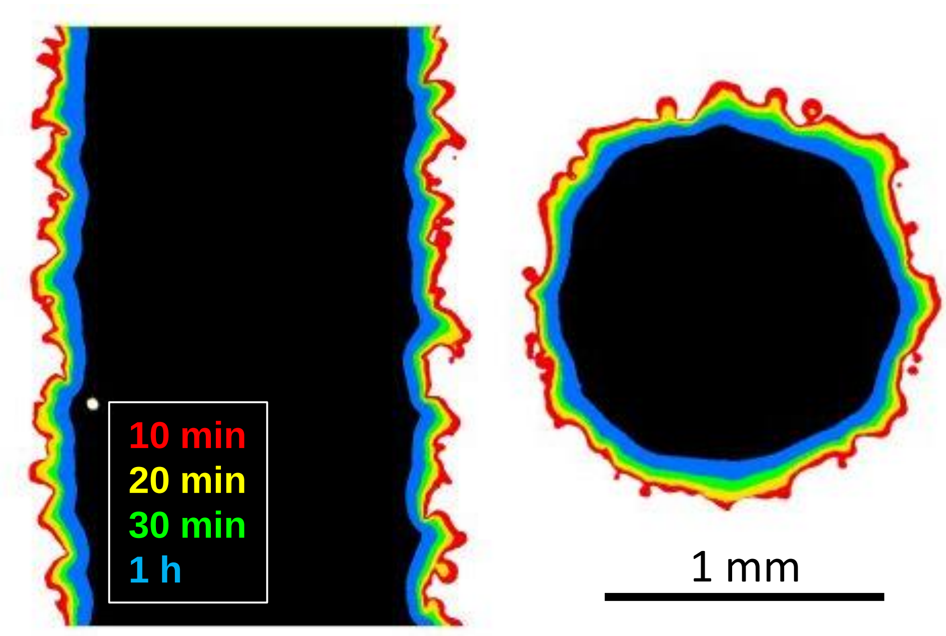
Chemical etchant:
3%HF, 13% HNO_3

Y.Y. Sun et al., *The Journal of The Minerals, Metals & Materials Society*, 2016

Surface post-treatments

Post-process treatment: chemical etching

Effect of etching time:

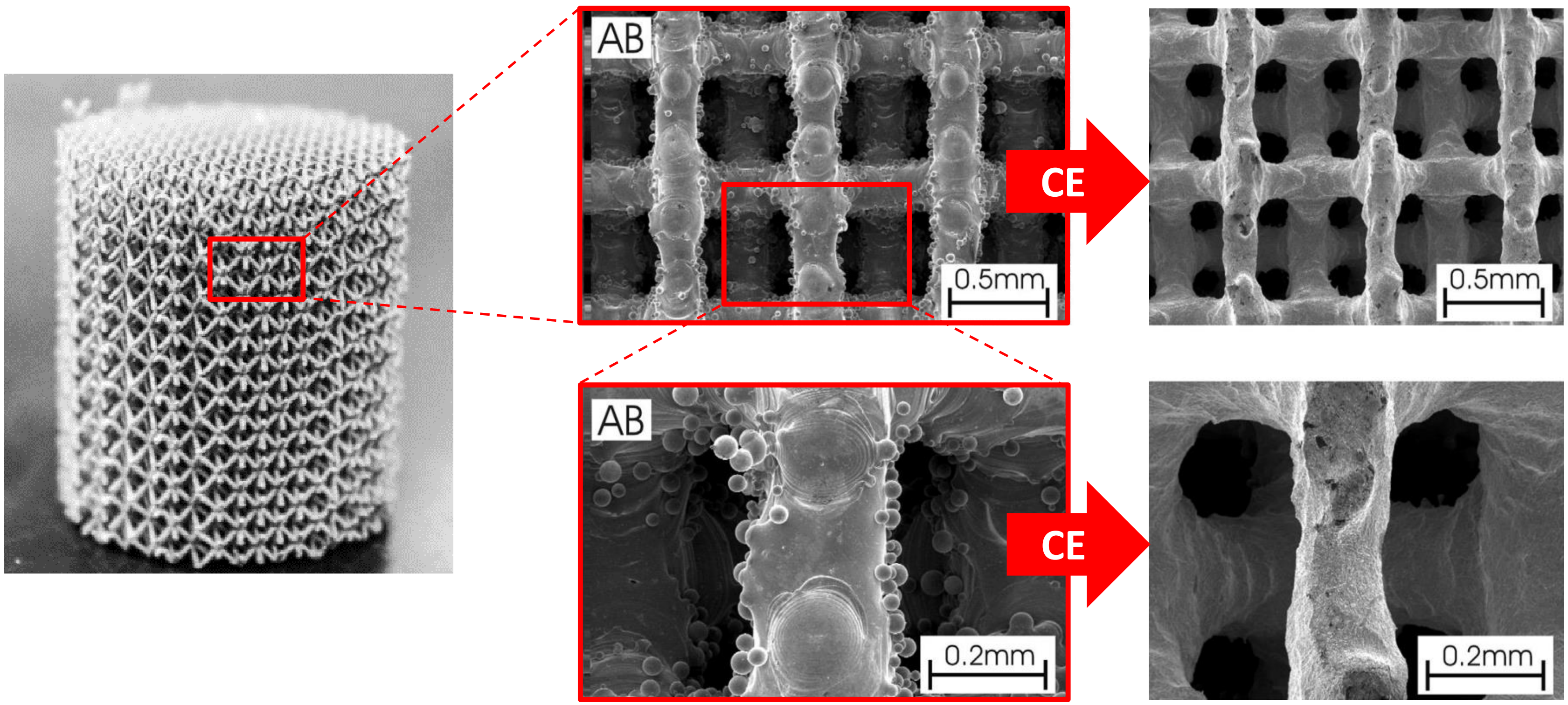


The grains of powder stuck to the surface are the first to be removed.

Surface post-treatments

Post-process treatment: chemical etching

Chemical etching is particularly adapted to intricate geometries (lattice structures)



B. Van Hooreweder et al., *Acta Biomaterialia*, 2017

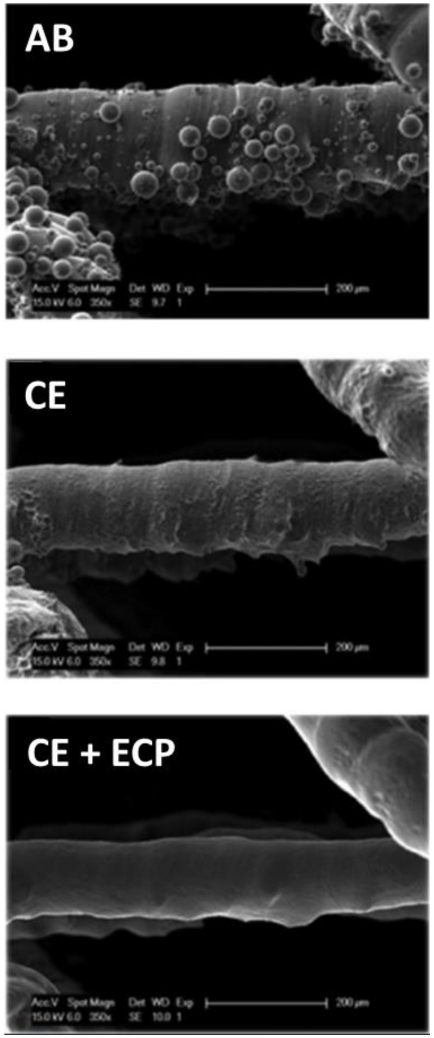
Surface post-treatments

Post-process treatment: combination of etching and electrochemical polishing

Chemical etching is mainly used to remove the partially melted or partially sintered particles on the surface.

Then, electrochemical polishing is used to make the surface of the part more uniform and smooth.

CE + ECP leads to a significant loss in strut thickness, which should be taken into account anticipatively in the design of the structure.



Pyka, Grzegorz, et al. "Surface modification of Ti6Al4V open porous structures produced by additive manufacturing." *Advanced Engineering Materials* 14.6 (2012): 363-370.

Post-treatments for microstructure and properties control

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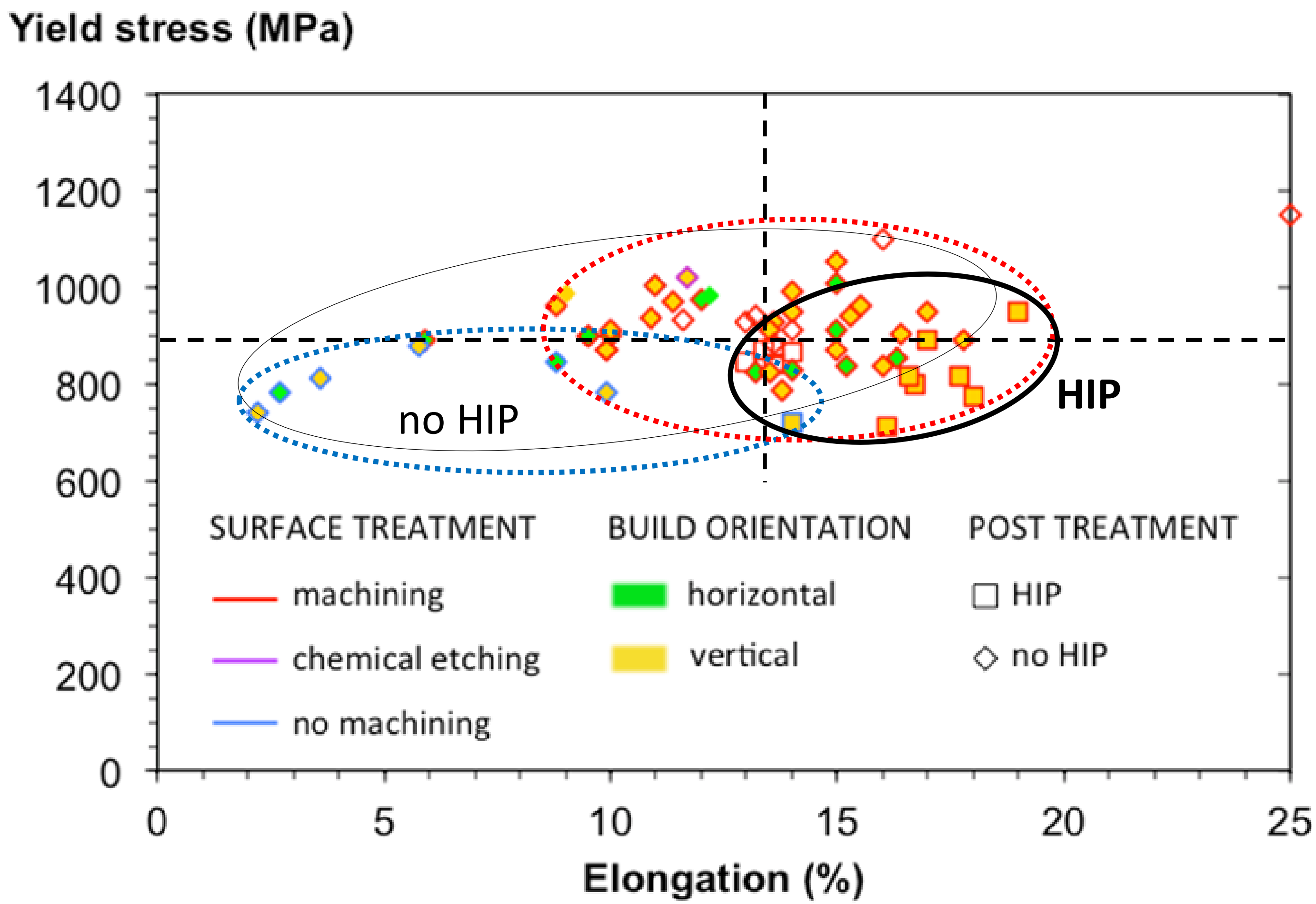
  - tensile
  - fatigue

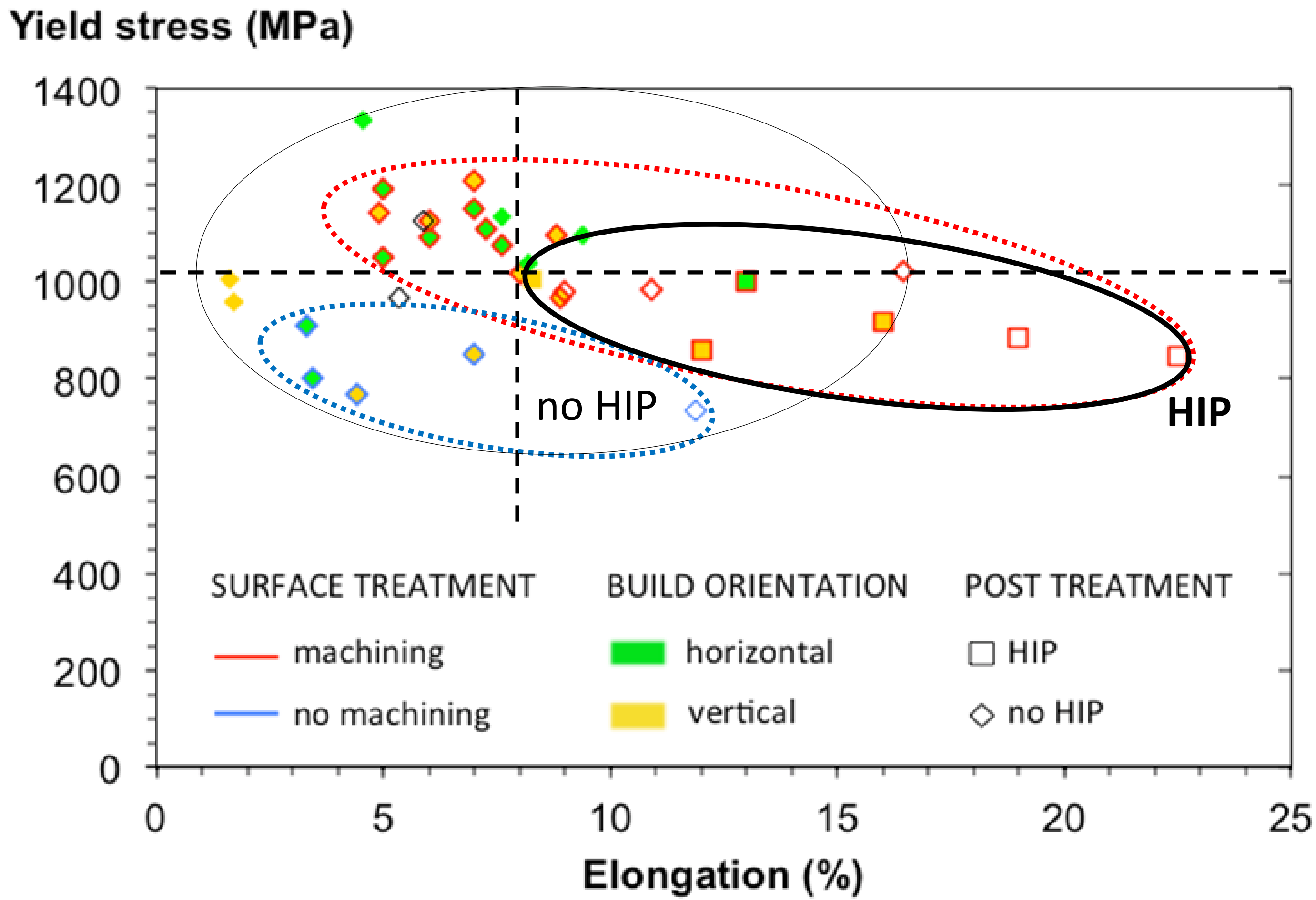
Effect of post-processing on tensile properties

EBM Ti-6Al-4V



Effect of post-processing on tensile properties

LPBF Ti-6Al-4V



Post-treatments for microstructure and properties control

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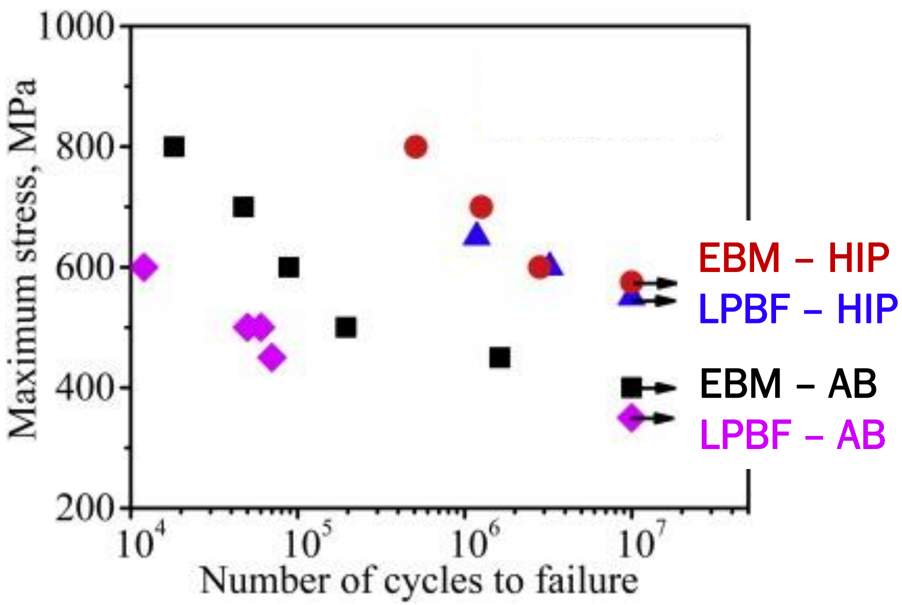
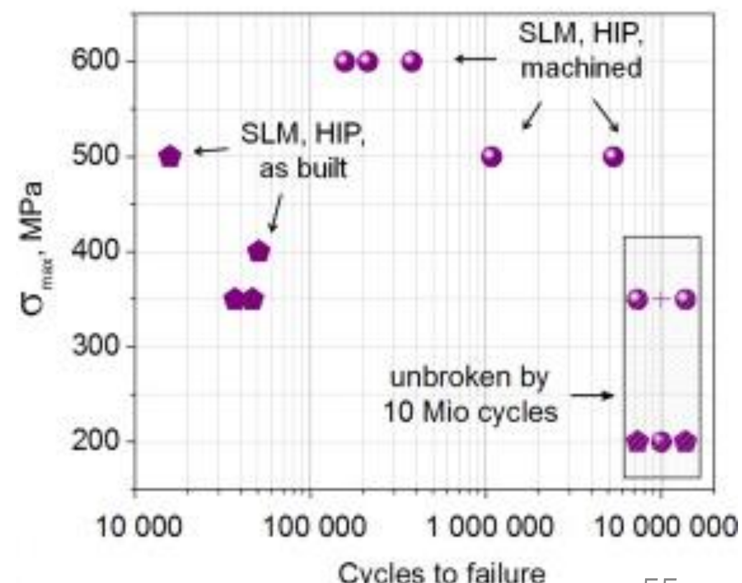
Effect of post-processing on fatigue properties of AM Ti-6Al-4V



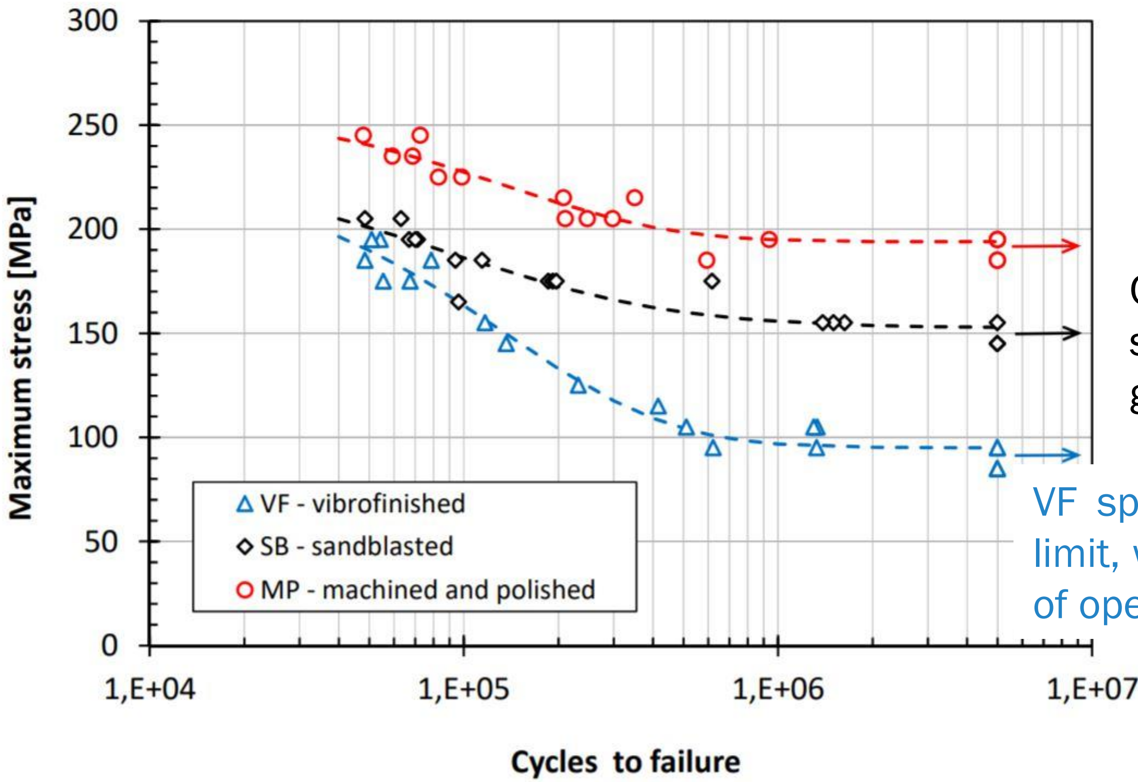
In as-built (AB) condition, LPBF parts have a lower fatigue life than EBM parts, due to high residual stresses.

HIP relieves residual stresses and removes critical defects from the bulk, increasing the fatigue life of LPBF and EBM parts.

Machining removes surface defects acting as crack initiators, further increasing the fatigue life of the material.

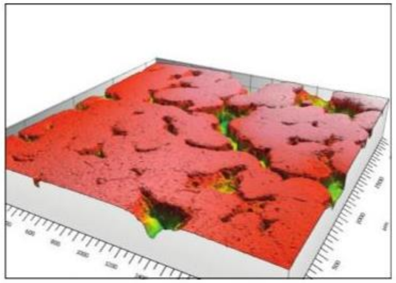


Effect of post-processing on fatigue properties of AM Ti-6Al-4V



Compressive residual stresses and strain hardening of the surface are generated during SB

VF specimens show the lowest fatigue limit, which is attributed to the presence of open surface and subsurface defects



Hamidi Nasab, Milad, et al. "Effect of surface and subsurface defects on fatigue behavior of AlSi10Mg alloy processed by laser powder bed fusion (L-PBF)." *Metals* 9.10 (2019): 1063.
Atzeni, Eleonora, et al. "Performance assessment of a vibro-finishing technology for additively manufactured components." *Procedia CIRP* 88 (2020): 427-432.