Low temperature martensitic decomposition in laser powder bed fusion produced Ti-6Al-4V

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December 2019

MSSA Conference
1 - 5 December 2019
CLUB MYKONOS • LANGEBAAN • WESTERN CAPE

FAKULTEIT INGENIEURSWESE
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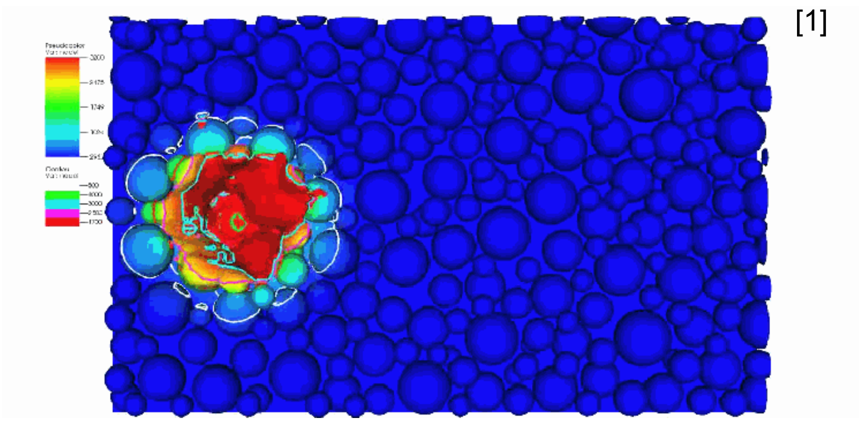
Background - Metal additive manufacturing

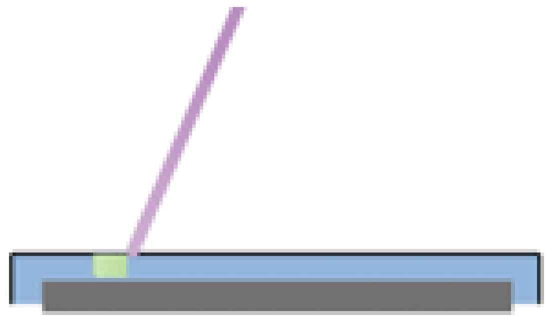
Additive **manufacturing** (“3d printing”)
Selective laser melting or **Laser Powder Bed Fusion**



[1]

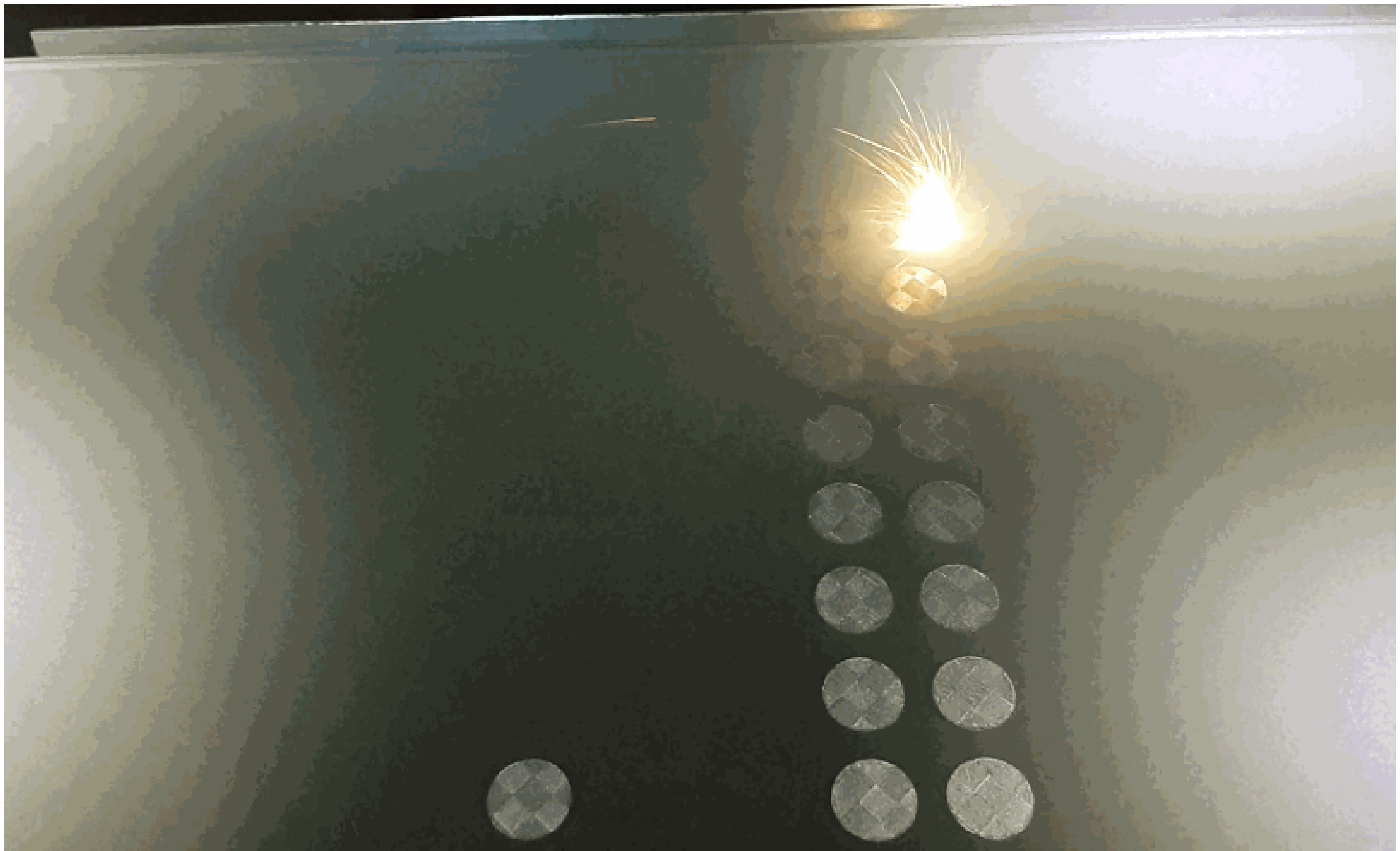
100 µm

[2]



1. Khairallah, S. A.; Anderson, A. T.; Rubenchik, A.; King, W. E. Laser powder-bed fusion additive manufacturing: Physics of complex melt flow and formation mechanisms of pores, spatter, and denudation zones. *Acta Materialia* **2016**, *108*, 36–45,
2. D. Raabe:3D Printing of Metals: Laser Additive Manufacturing (LAM) via Selective Laser Melting (SLM) and Laser Metal Deposition (LMD) [ONLINE]: http://www.dierk-raabe.com/laser-additive-manufacturing/

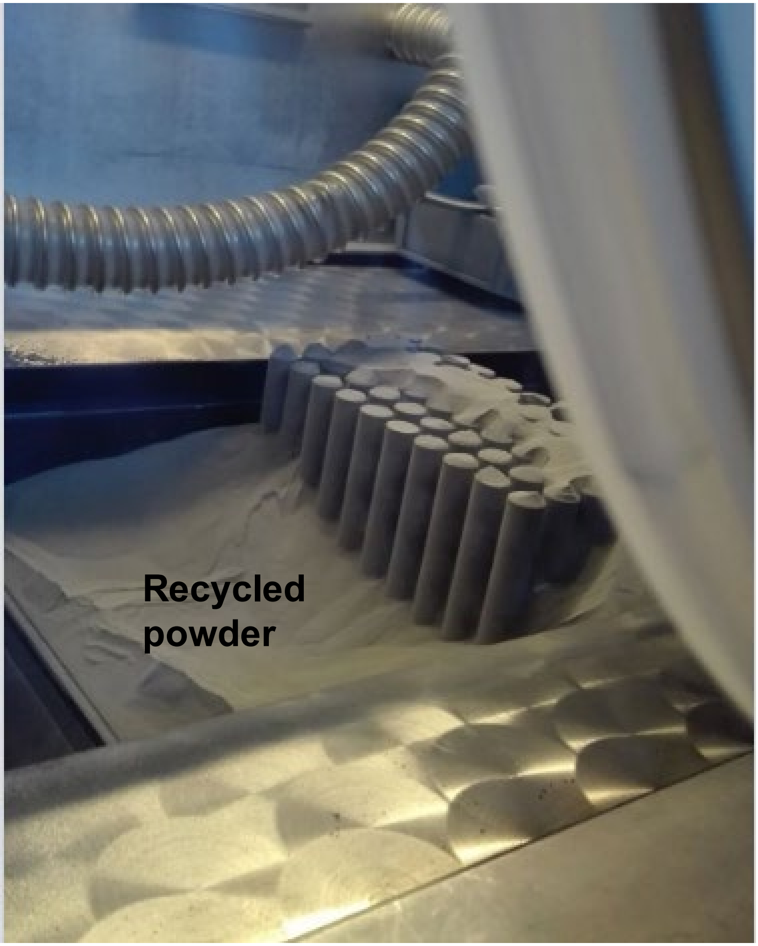
Background - Metal additive manufacturing



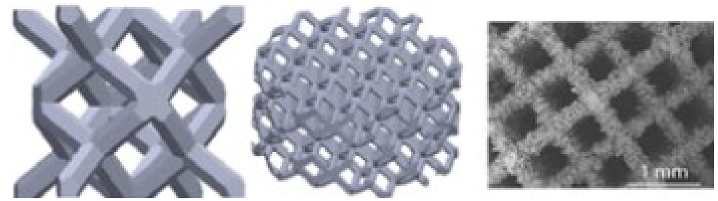
Argon flow

Background - Metal additive manufacturing

One week and 3 434 layers later



Why MAM, why Ti6Al4V, why PhD?



[1]

[2]

1. Advantages of additive manufacturing

- Manufacturing flexibilities (complex shapes)
- Environmentally sustainable (no material waist)
- Lead time reduction
- Manufacturing democratisation (customisation)

2. Advantages of Ti-6Al-4V

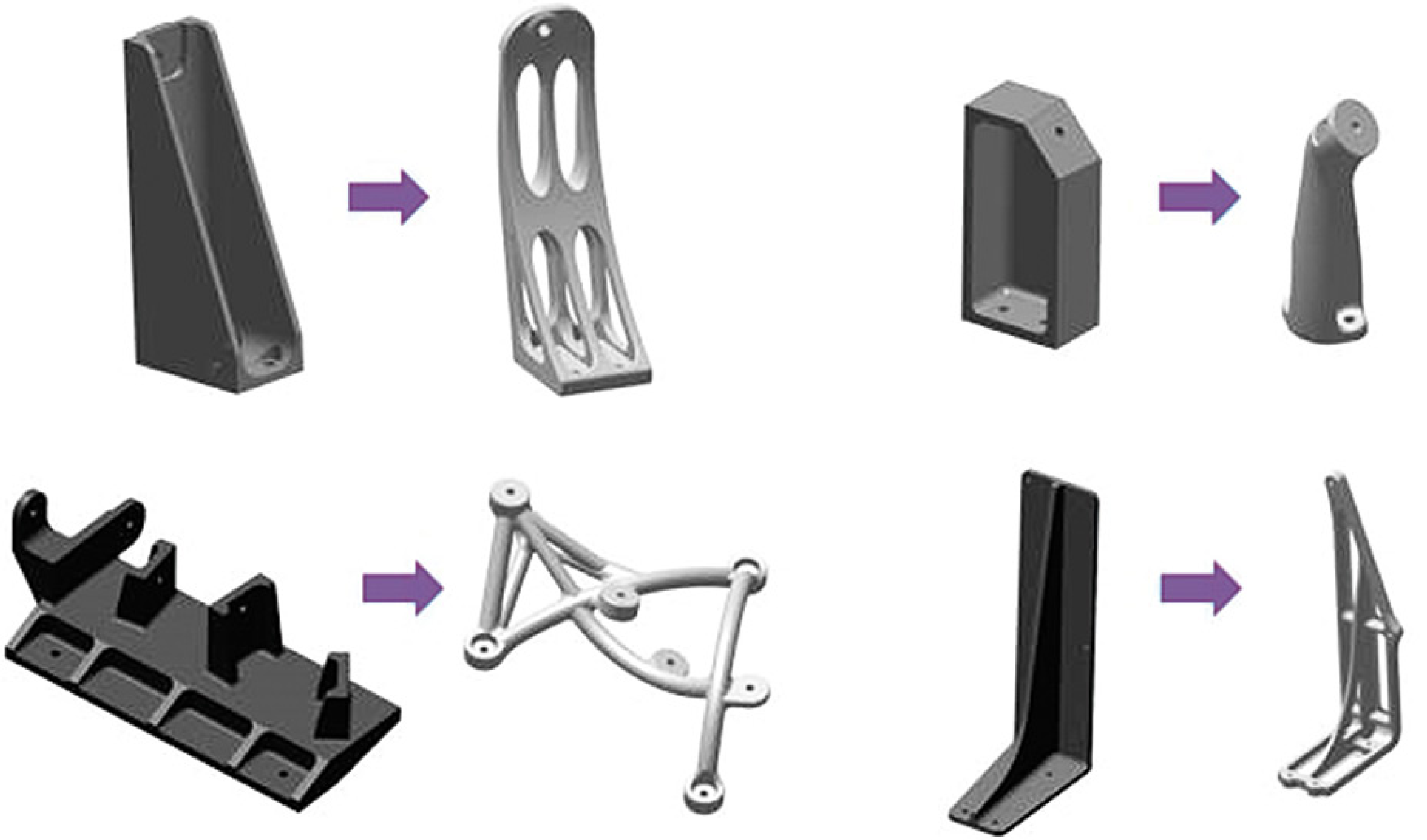
- Biocompatible (medical implants)
- Excellent mechanical properties:
-    Titanium is 30% stronger than steel, & 50% lighter (cost saving)
- Moderate temperature corrosion resistant ~ 400/500°C
- Example: Boeing saving $2-3 million per plane (2017)

3. Concern over achievable mechanical properties (quality)

- Martensitic microstructure -> poor ductility
- Poor fatigue life
- Residual stress
- Porosity

[1] W. Hoffmann, F.Schlottig, M.Mertmann, M. de Wild, D. Wendt, I. Martin, The interplay between NiTi-SMA and human bone marrow-derived mesenchymal stromal cell, Proceeding p. 46-47 of the 4th International Symposium Interface Biology of Implants IBI, 9.-11. May 2012, Warnemünde/Rostock (Germany).
[2] R. Schumacher, M. de Wild, E. Schkommodau, D. Hradetzky, Massgeschneiderte Knochenimplantate aus dem 3D-Drucker,BaZ-Sonderbeilage "Life Sciences" vom 12. Mai (2012).

Examples

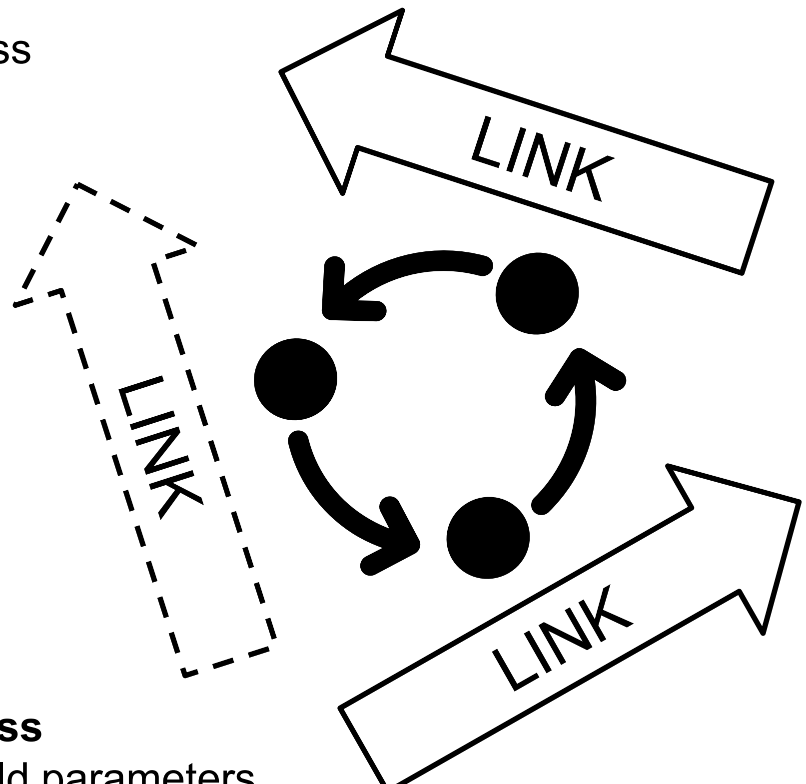


Aston, R. (2018). *Boeing: 3D printing done right*. [online] Boeing.com. Available at: https://www.boeing.com/features/innovation-quarterly/nov2017/feature-thought-leadership-3d-printing.page [Accessed 14 Nov. 2018].

PhD technical background

Knowledge of process-structure-property links

- **Ductility**
- Fracture toughness
- Fatigue
- **Anisotropy**



- Porosity
- **Microstructure**
- Residual stress
- Surface roughness

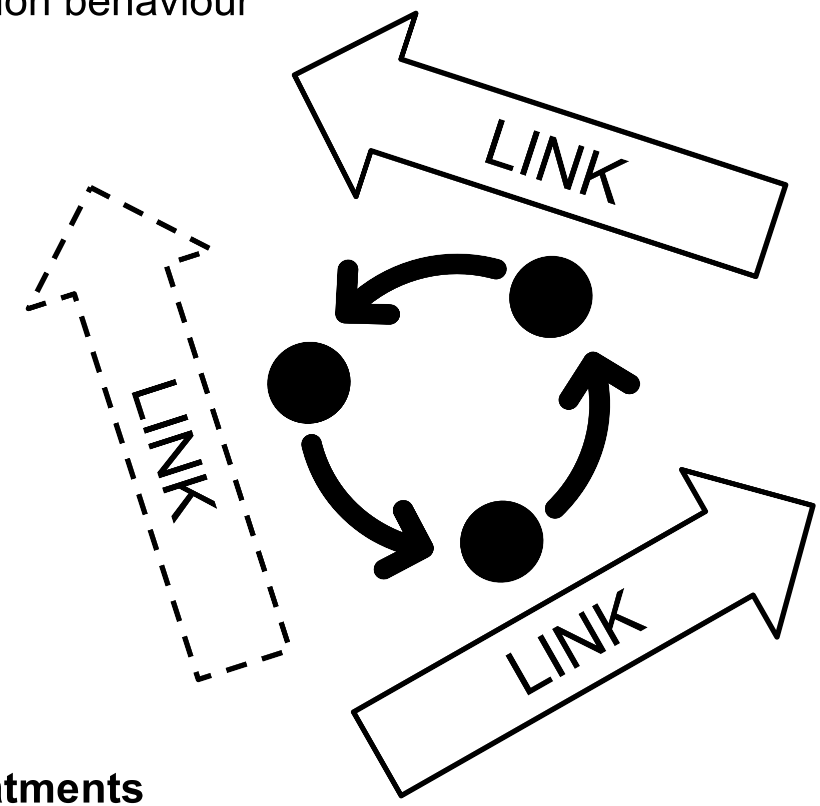
Build process
- LPBF build parameters
- (laser power, scan speed etc)

Post-process
- Thermal treatments
- Hot Isostatic Pressing

PhD technical background

Knowledge of process-structure-property links

- Tensile deformation behaviour



- **Microstructure**
- **Residual stress**

- **Thermal treatments**

Wrought Ti-6Al-4V vs LPBF Ti-6Al-4V mechanical properties

Wrought Ti-6Al-4V

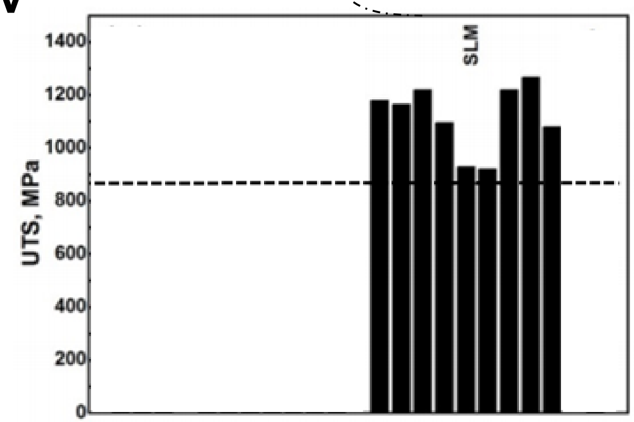
Solution-treating temperature		Room-temperature tensile properties(a): Tensile strength		Room-temperature tensile properties(a): Yield strength (b)		Elongation in 4D(c),
°C	°F	MPa	ksi	MPa	ksi	%
845	1550	1025	149	980	142	18
870	1600	1060	154	985	143	17
900	1650	1095	159	995	144	16
925	1700	1110	161	1000	145	16
940	1725	1140	165	1055	153	16

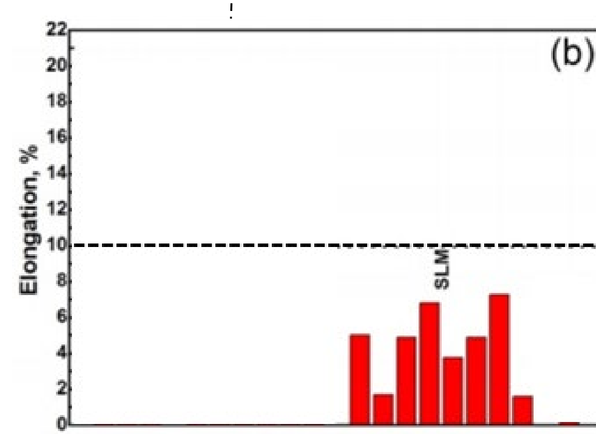
(a) Properties determined on 13 mm (½-in.) bar after solution treating, quenching, and aging. Aging treatment 8 h at 480 °C (900 °F), air cool. (b) At 0.2% offset. (c) D, specimen diam

[1]

LPBF-produced Ti-6Al-4V

ASTM F2924–14 ---→



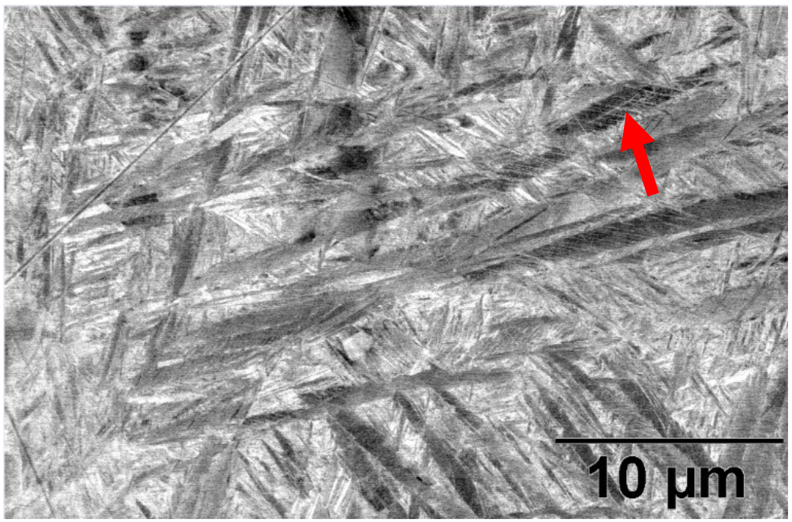


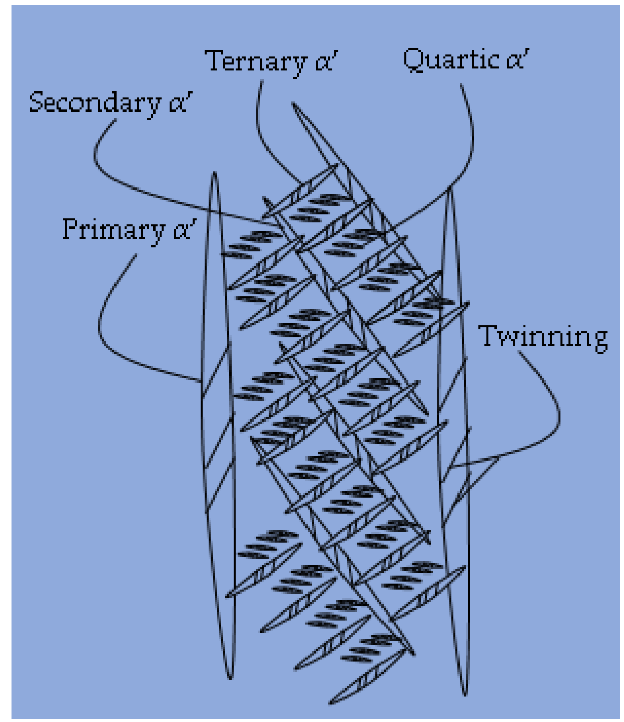
[2]

[1] Donachie, M. 2000. *Titanium A Technical Guide*, Ohio.
[2] M. Yan and P. Yu, "An Overview of Densification, Microstructure and Mechanical Property of Additively Manufactured Ti-6Al-4V — Comparison among Selective Laser Melting, Electron Beam Melting, Laser Metal Deposition and Selective Laser Sintering, and with Conventional Powder," in *Sintering Techniques of Materials*, INTECH, 2015, pp. 76–106.

As-built Ti-6Al-4V martensite (α’)

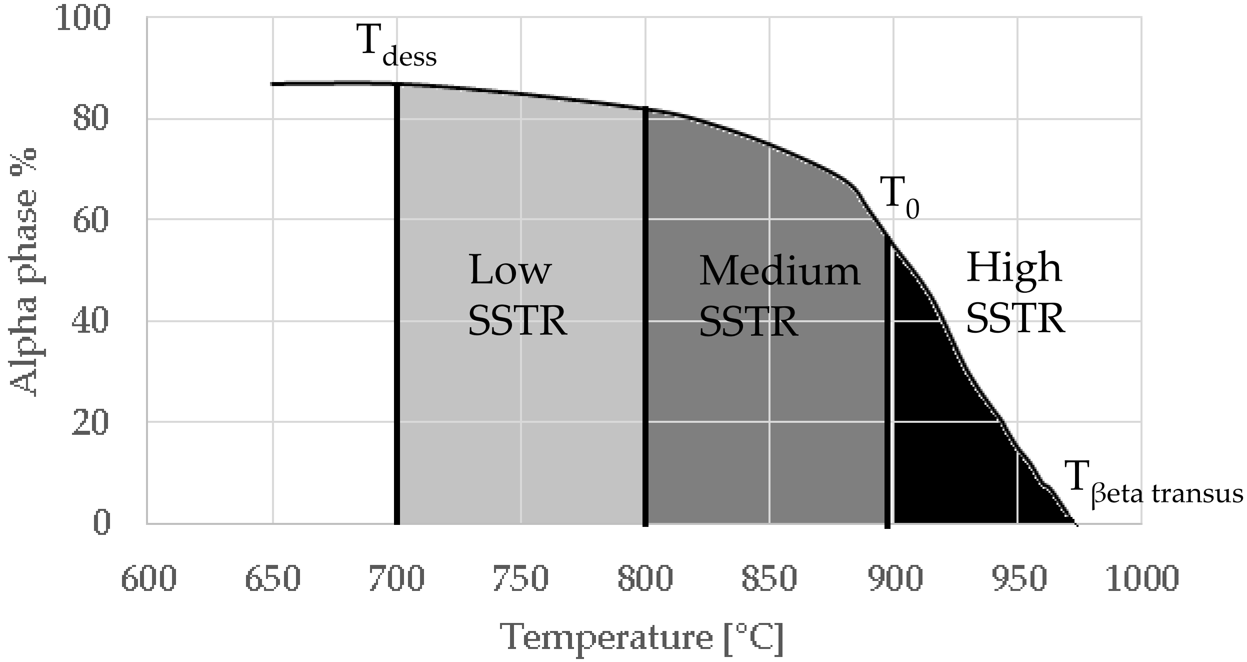
Single phase hierarchical martensite structure (HCP)
Form through fast cooling
Large range in grain size
Dislocations and twinning

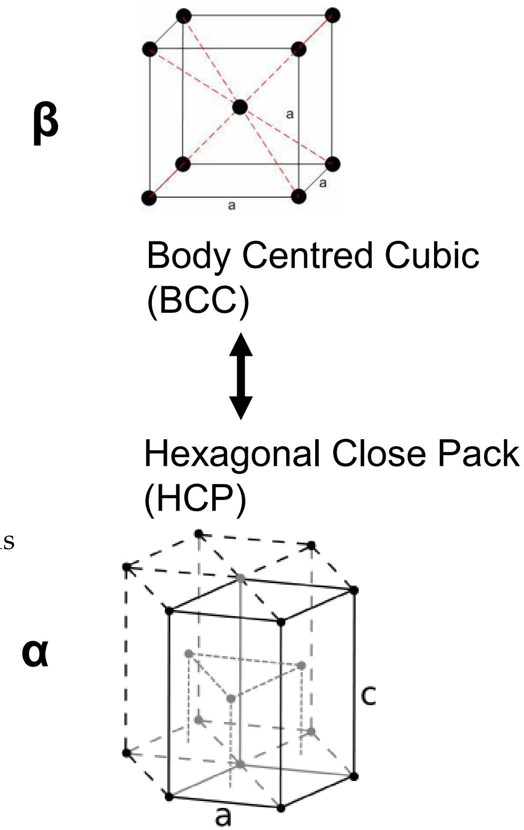




Metallurgy and heat treatments

- **Duel-phase α+β**
- **Decomposition from α’ → α+β (more ductile)**
- **Loss of strength due to α grain growth**





3. Optimal - Bi-modal (α + α’)

- **Unique contribution - fragmentation and globularisation of alpha grains**

materials

MDPI

Article

Selective Laser Melting Produced Ti-6Al-4V: Post-Process Heat Treatments to Achieve Superior Tensile Properties

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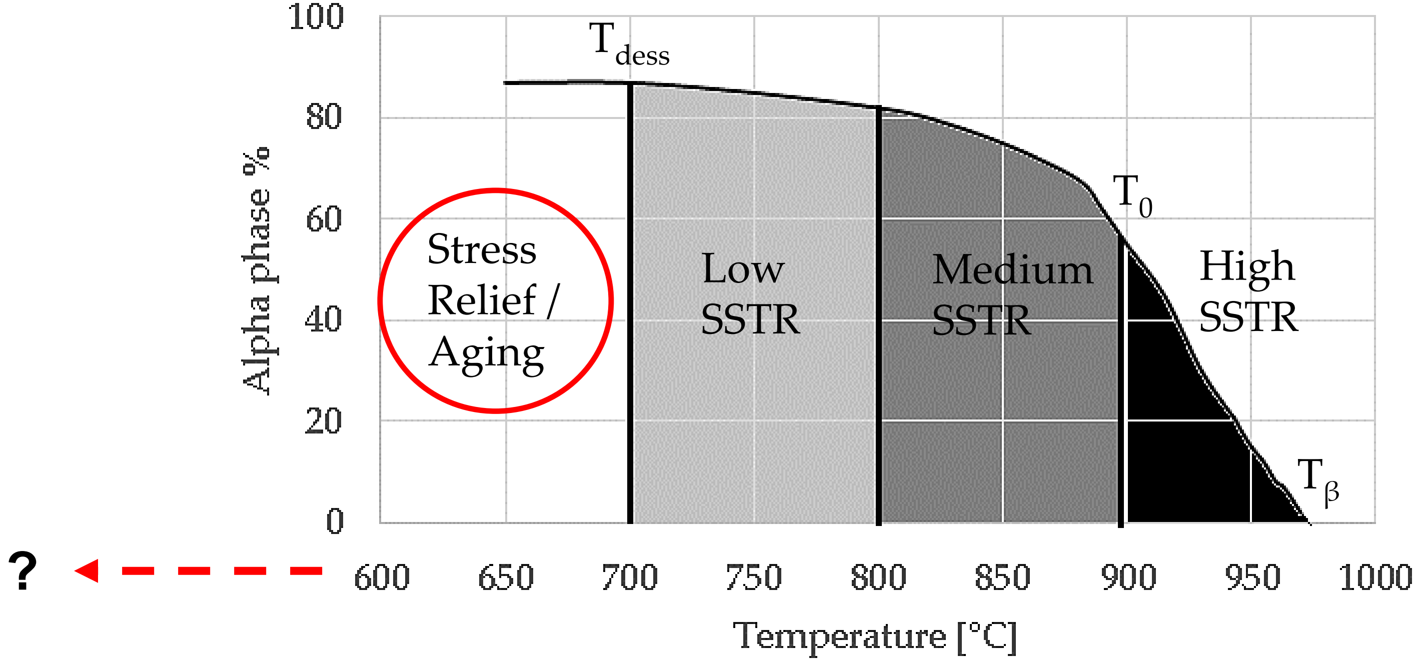
Received: 9 November 2017; Accepted: 15 December 2017; Published: 17 January 2018

Aim of current work



Motivation and aim

Motivation & aim

1. Improve material strength by annealing at lower temperature, while still relieving residual stress.

2. Apply knowledge to base-plate pre-heating strategies.

3. Key technical questions:

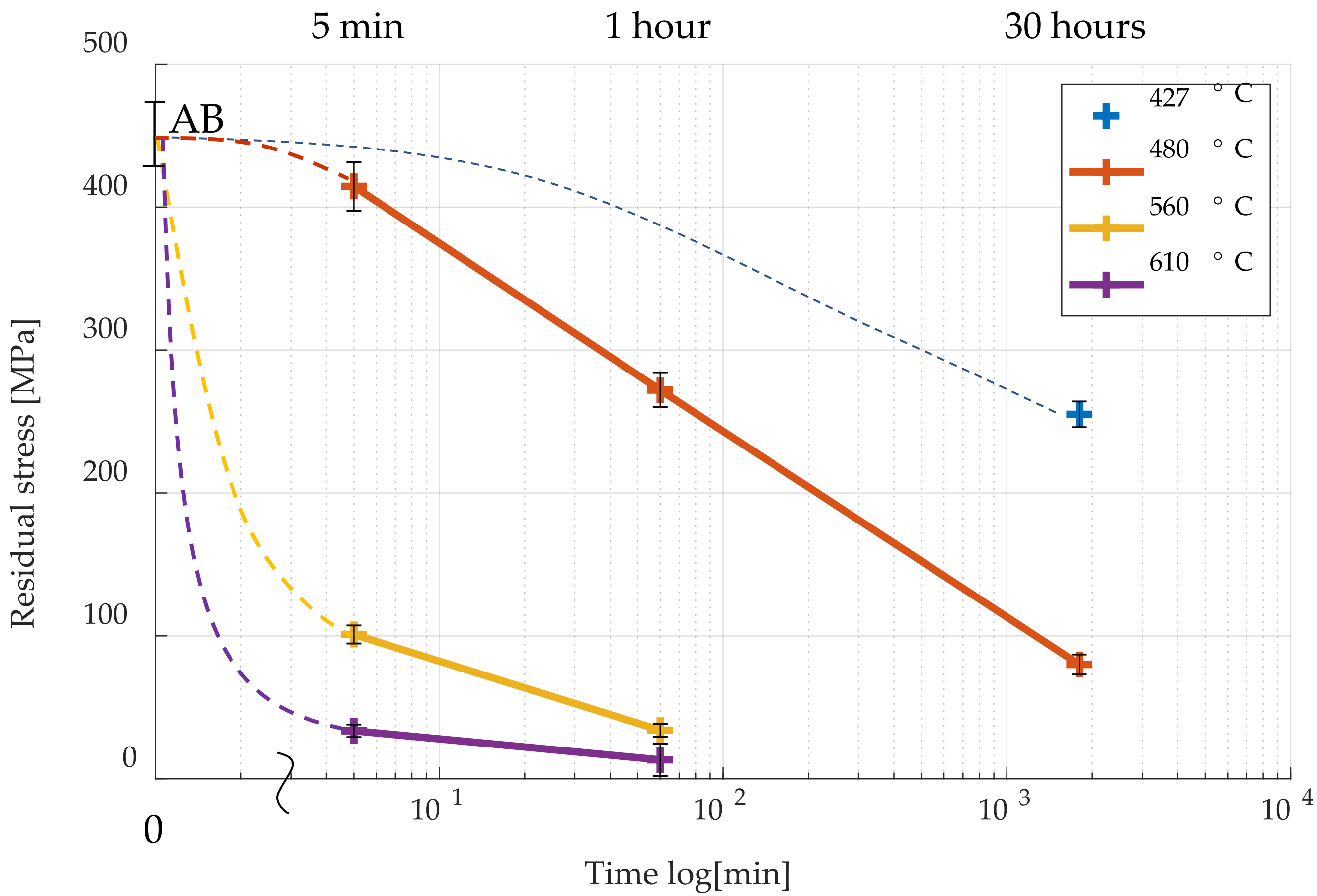
- ❑ What is the nature of martensite decomposition at **low** temperatures?
- ❑ How does it influence strength and ductility?
- ❑ Does Ti_3Al form?

Experimental approach

1. Samples
   - Ti6Al4V ELI powder
   - EOS M280 machine standard build parameters
   - “Dog-bone” shapes (machined post-process)
   - Vertical orientation
   - **Reference**: 1000 °C 2 hours, furnace cooled (Stable α+β lamellar structure)
2. Temperatures: (**427, 480, 560, 610 °C**)

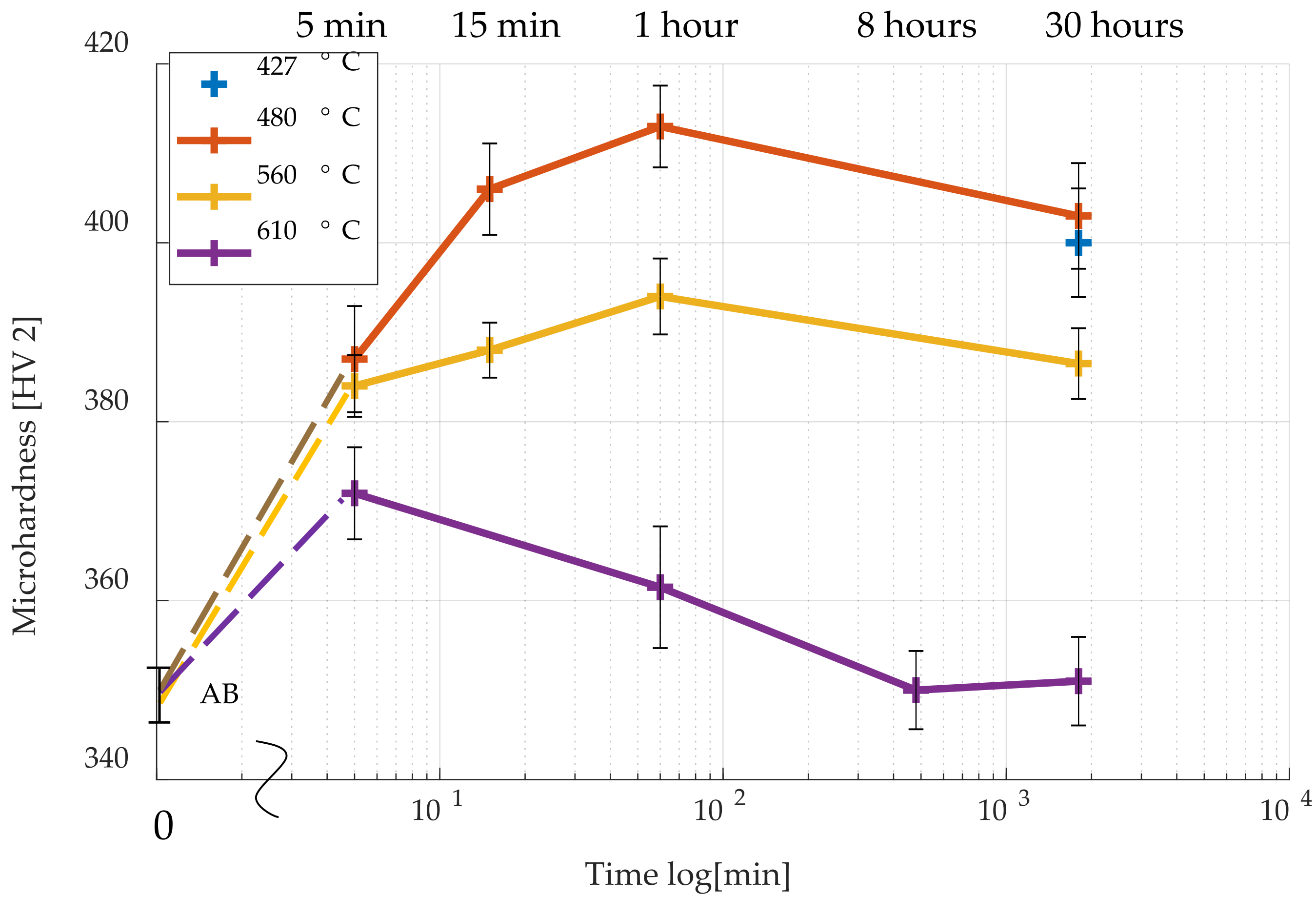
	Experiment/measurement					
	Residual stress	**Hardness**	**SEM**	**STEM**	**Rietveld Refinement**	**Tensile test**
As-built	✓	✓	-	-	✓	✓
Reference	-	✓	✓	-	✓	-
Hold time						
5 min	all	all	-	480	480	-
15 min	-	480, 560	-	-	480	-
1 hour	all	all	-	480	480	480, 560, 610
8 hours	-	610	-	-	-	610
30 hours	427, 480	all	all	480	all	-

Residual stress profiles



AB – As-built

Microhardness profiles

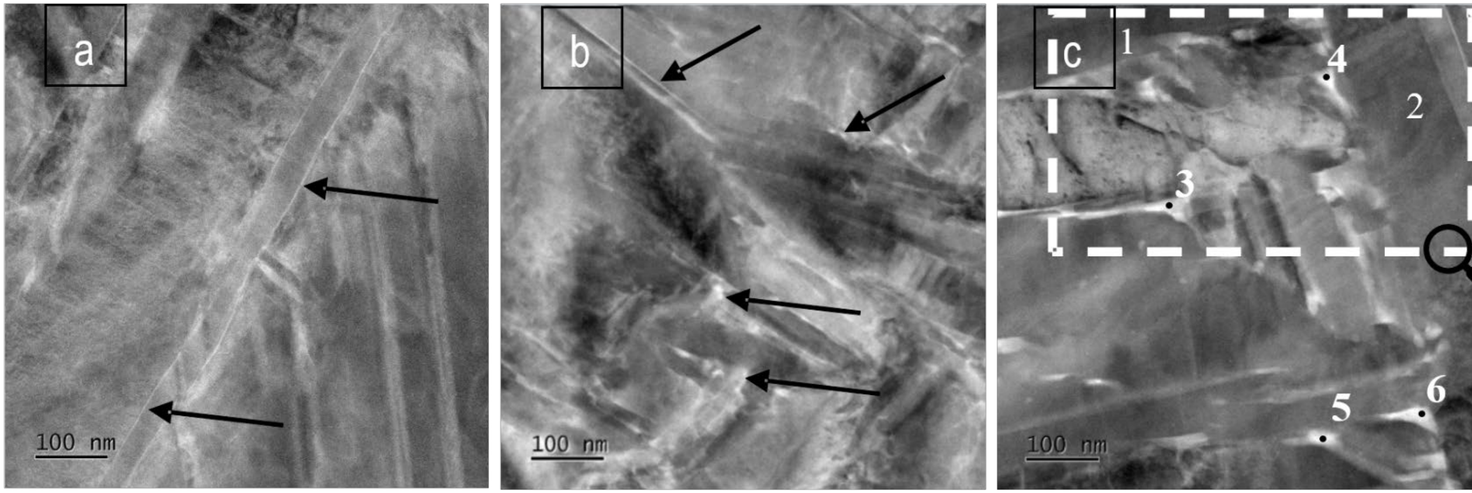


AB – As-built

Early stages of phase transformation

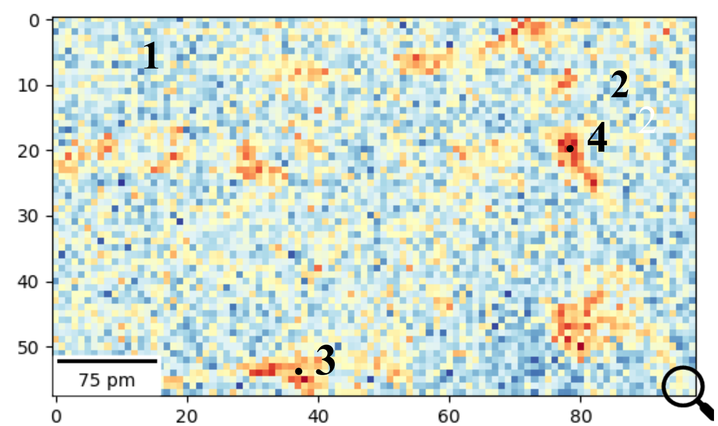
480 ºC 5 min 1 hour 30 hours

HAADF-STEM



	Weight % (σ)		
	Ti	**Al**	**V**
1	89.4 (0.5)	**4.3 (0.3)**	4.3 (0.4)
2	89.5 (0.2)	**4.3 (0.1)**	6.2 (0.2)
3	81.9 (0.2)	4.5 (0.1)	**13.6 (0.1)**
4	78.5 (0.2)	3.8 (0.2)	**17.8 (0.1)**
5	80.7 (0.2)	4.3 (0.2)	**15.0 (0.1)**
6	79.7 (0.2)	4.2 (0.1)	**16.1 (0.1)**

Reference sample (stable α+β)			
α	91.2 (0.2)	**6.8 (0.1)**	2.2 (0.1)
β	80.1 (0.2)	2.5 (0.1)	**17.4(0.1)**



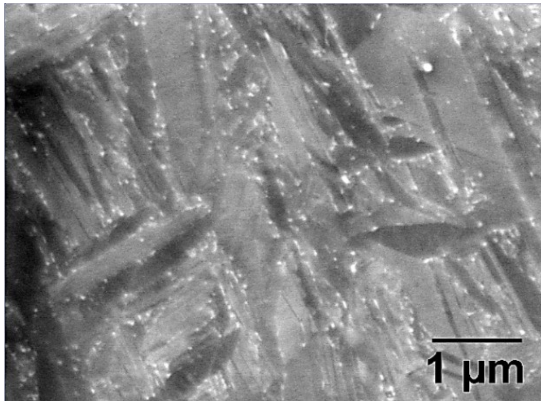
Electron energy loss spectroscopy (EELS)

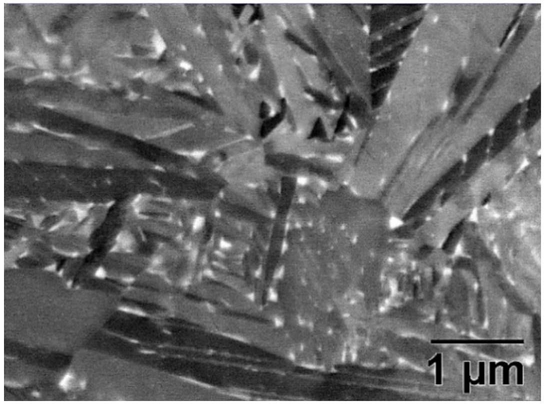
Micrographs and X-ray diffraction

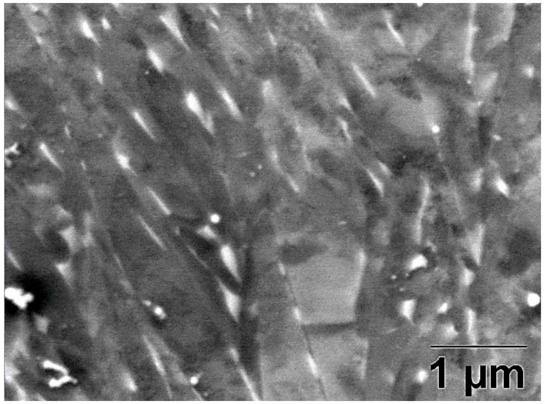
30 hours

480 ^{o}C | 560 ^{o}C | 610 ^{o}C

BSD-SEM

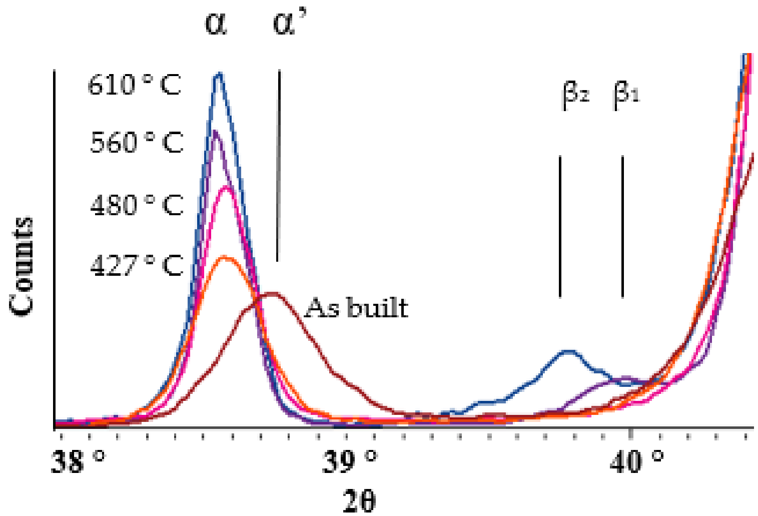




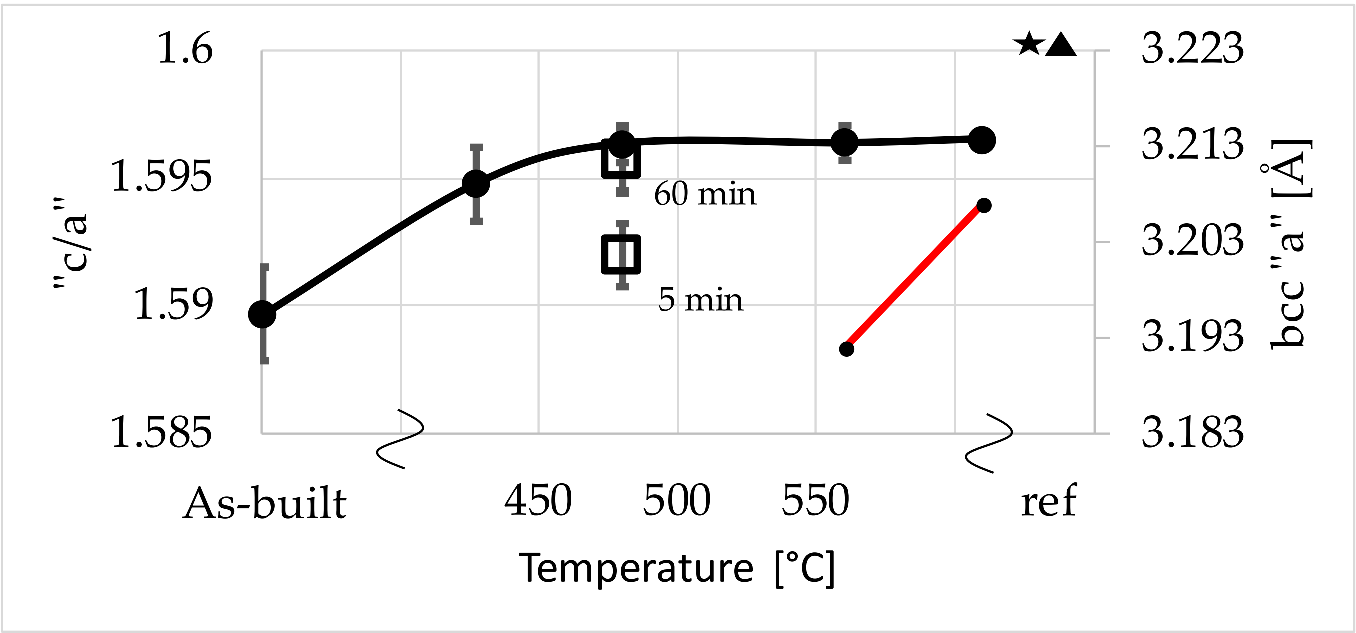


Peak shift
Peak intensity changes

XRD Rietveld:
Beta phase fraction
560 ^{o}C 30 hours = ~4.5 %
610 ^{o}C 30 hours = ~6 %
Reference = **~10.2 %**

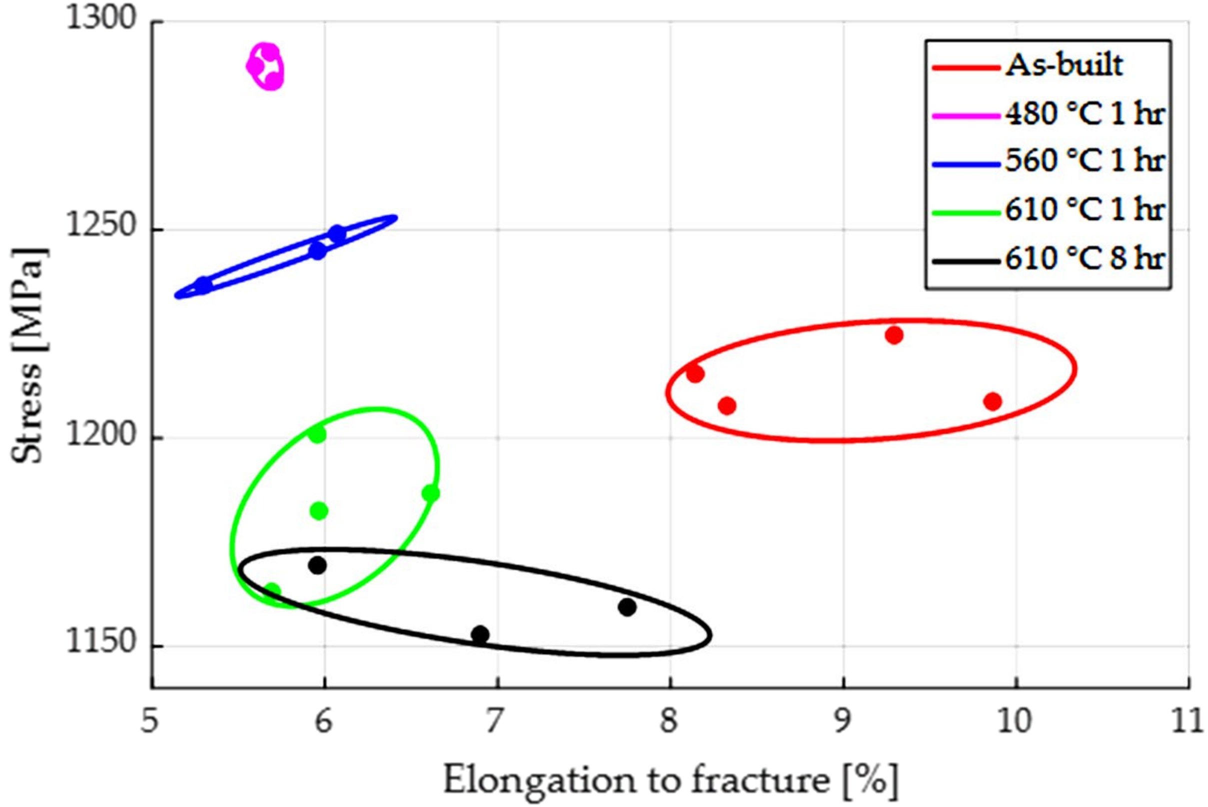


Lattice parameters transformation



"●" – 30 hour anneal, "□" – transitional times, "★" – reference *hcp*, "▲" – reference *bcc*

Tensile tests



Discussion

1. **Precipitation formation & phase transformation**
- Nucleation and growth of β-phase precipitates
- Spinodal decomposition (Atom probe tomography)
- Incomplete phase transformation

2. **Non-classical hardening**
- No Ti_3Al – due to low aluminium content in alpha phase
- α-β grain boundary strengthening
- Softening after 30 hours – beta phase increase, grain growth and lattice strain relaxation

Conclusion

In-depth study of initial martensite decomposition – embrittlement / strengthening

Non-classical hardening

Incomplete phase transformation (lattice parameter comparison to reference sample)

Recommendations:

1. **Stress relief:**
Above 560 °C for 1 hour or more (90 % stress relief)
OR 30 hours 480 °C (in-situ)

2. **Strengthening**
480 °C for 1 hour.
Use in addition to bi-modal (α+α’)

3. **Ductility improvement**
Annealing above 610 °C for longer than 8 hours.

Acknowledgments

Funding: Department of Science and Innovation
CSIR's Collaborative Program for
Additive Manufacturing (CPAM) project

STEM – Nelson Mandela University (CHRTEM)
Johan Westraadt

SEM – CAF (Stellenbosch University)

NECSA – Residual stress

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