ADDITIVE 4 BIOMEDICAL

UNIVERSITÀ DEGLI STUDI FIRENZE
DIEF DIPARTIMENTO DI INGEGNERIA INDUSTRIALE

October 17th–18th, 2022

PSEUDOELASTICITY OF NITI PARTS MANUFACTURED BY LASER POWDER BED FUSION: HOW FAR WE ARE FROM THE CONVENTIONAL MANUFACTURING ROUTE FOR MEDICAL DEVICES?

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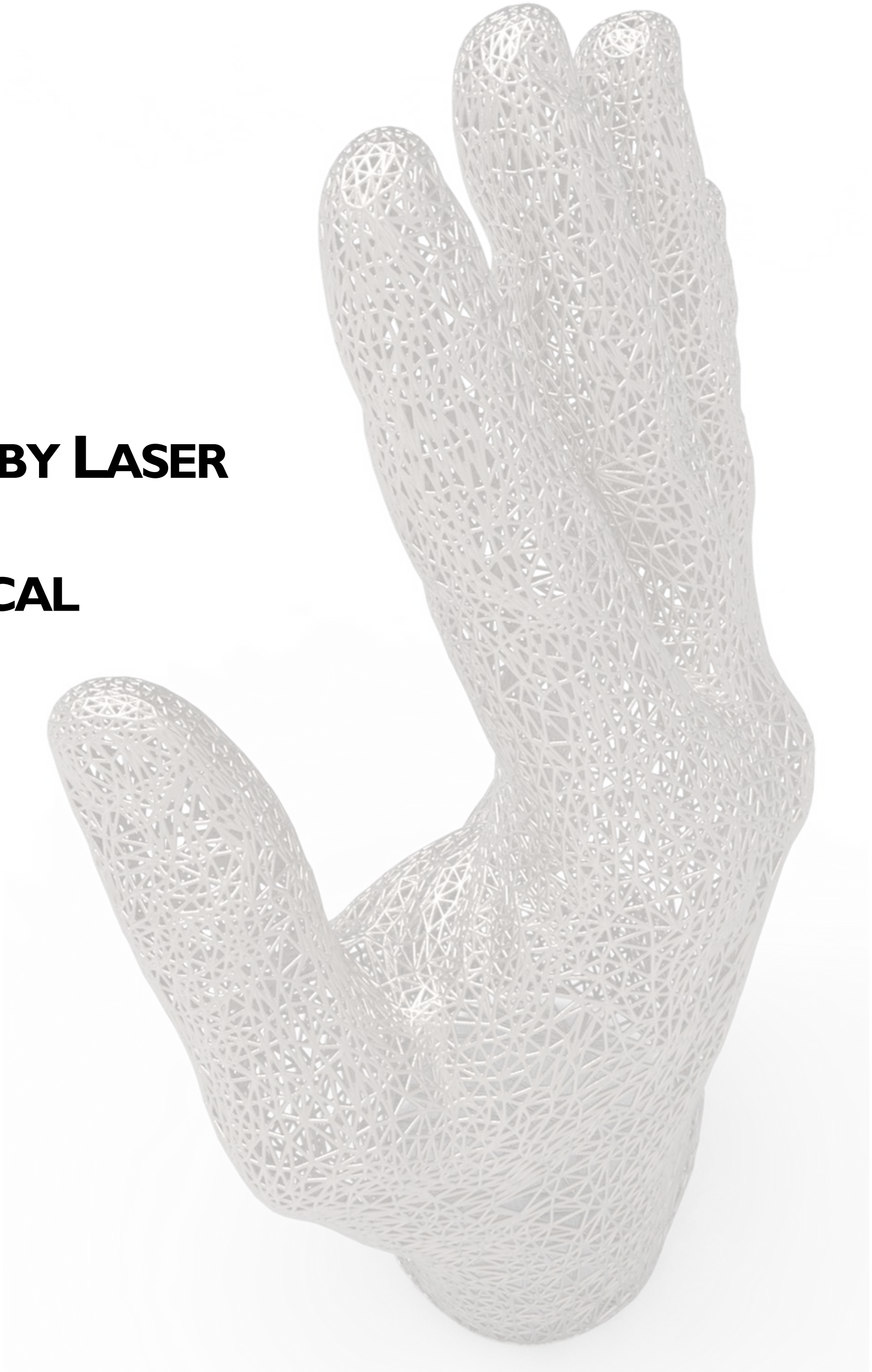
SC 09/A3 CoCo AM Additive Manufacturing

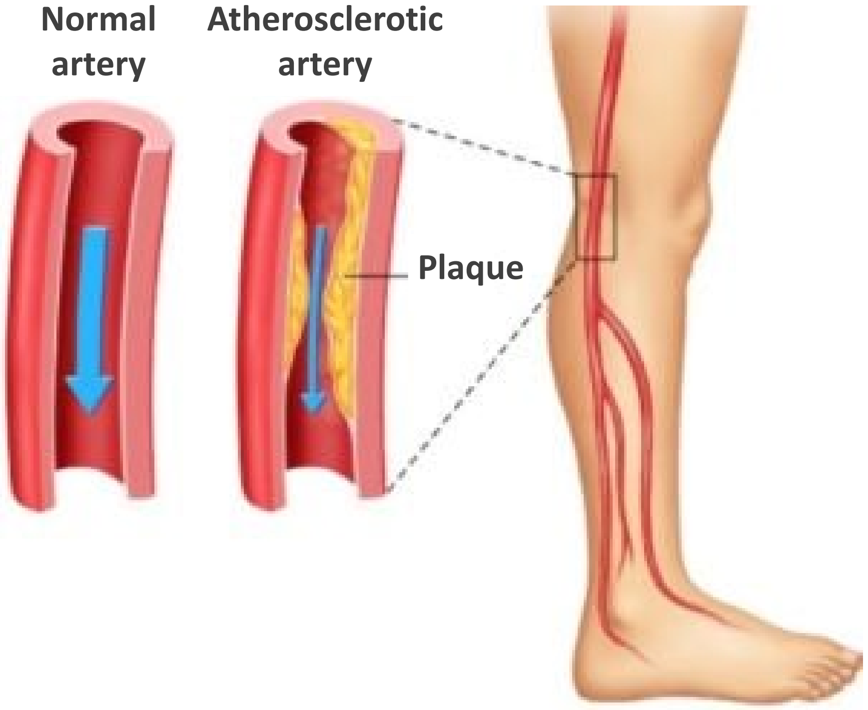
ASSOCIAZIONE NAZIONALE DISEGNO E METODI

COMET

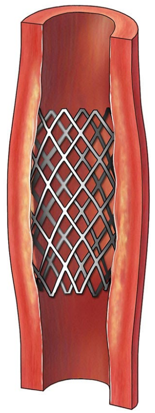
aias



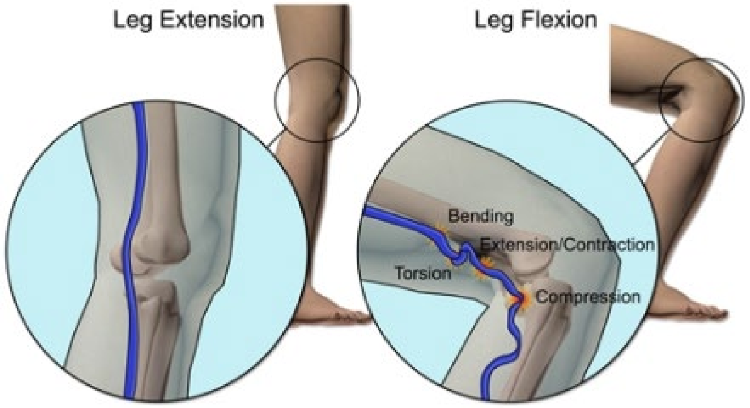
INTRODUCTION

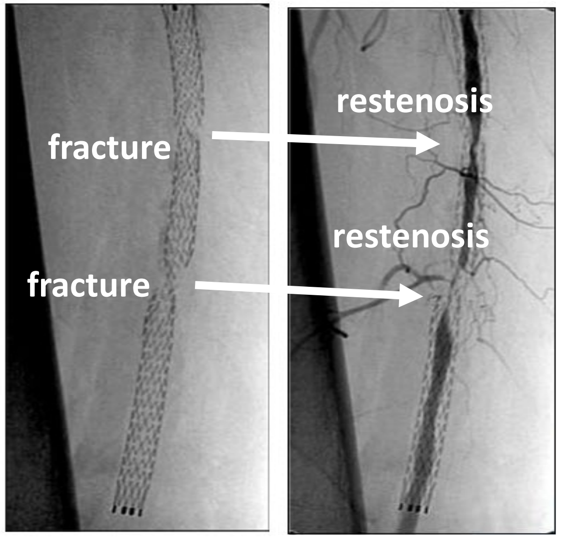


Gold standard
→ stenting



<u>1 million gaits/year</u>
stent **fatigue failure** and **severe consequences**

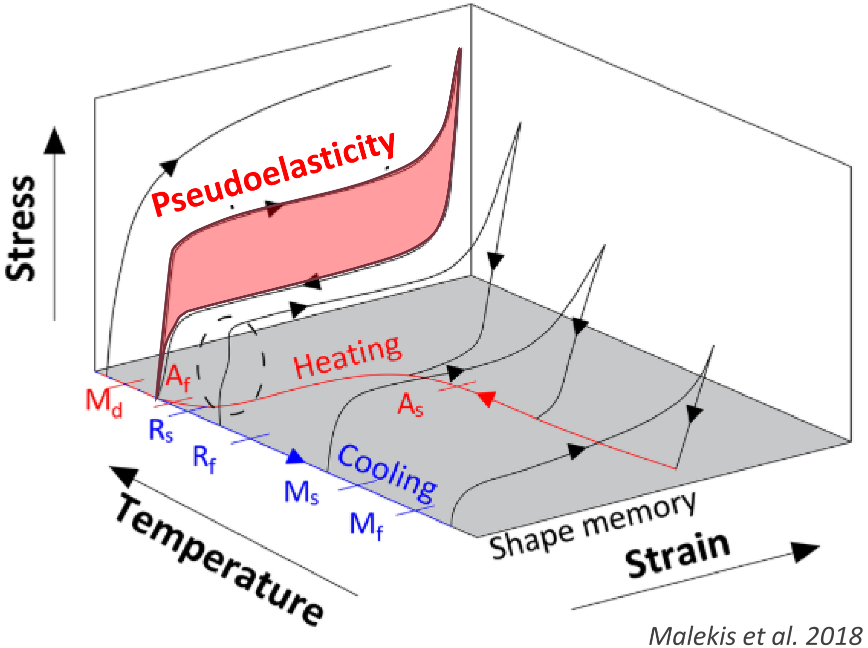




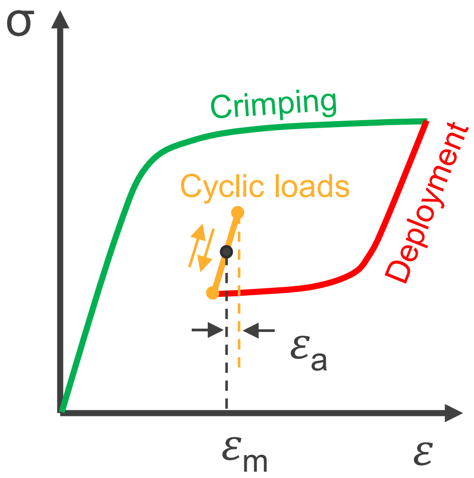
Scheninert et al. 2005

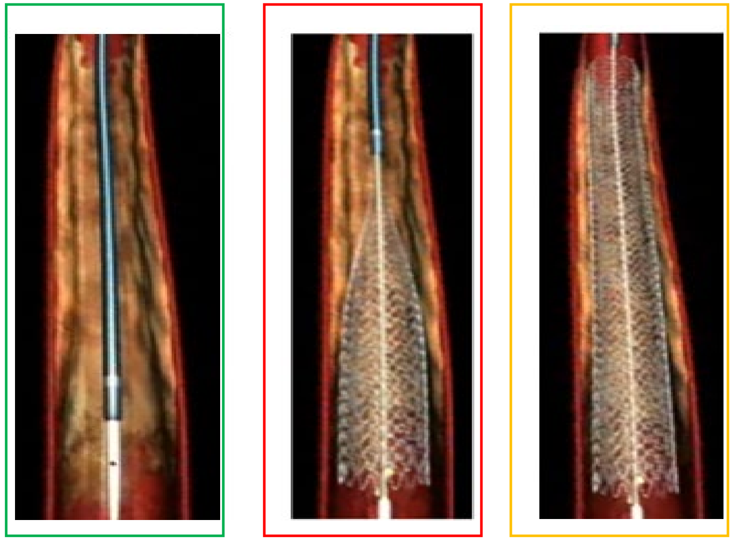
INTRODUCTION

Ni-Ti alloys
→ *gold standard* for peripheral stenting



Malekis et al. 2018





Crimping: diameter reduction to fit on the catheter

Deployment: self-expansion into the vessel

Cyclic loads: movements due to gait

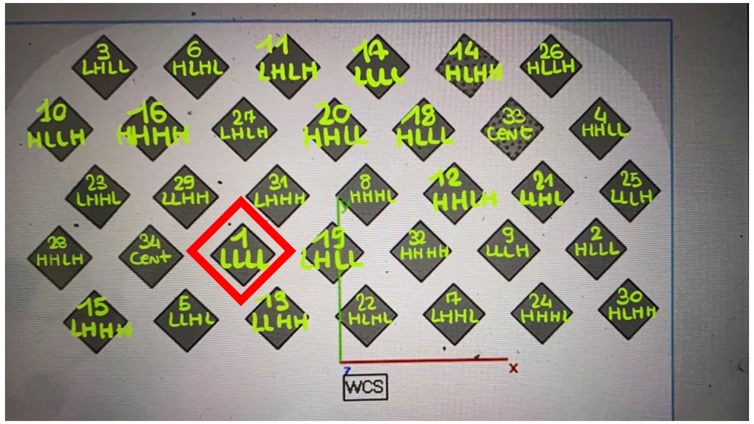
TRADITONAL MANUFACTURING APPROACHES vs AM

- The traditional manufacturing rout for NiTi stents consists in obtaining a tubular geometry and then laser-cutting to the final shape
- Such manufacturing strategy enables to obtain a **texturized material** with enhanced mechanical/functional properties
- In principle, **Additive Manufacturing (AM)** could enable more flexibility in the final geometry
- However, the strict regulations for bio-medical applications prevents the adoption of AM process
- NiTi alloy behavior strongly depends on chemical composition and heat treatment
- AMed NiTi requires further efforts for reaching the best functional performances offered by the traditional manufacturing routes
- In this presentation, we investigate the functional properties of AMed NiTi
- All the specimens were produced by means of a Renishaw AM250 industrial LPBF system

Carlucci, G., et al. "Building Orientation and Heat Treatments Effect on the Pseudoelastic Properties of NiTi Produced by LPBF." Shape Memory and Superelasticity (2022): 1-13.

FULL DENSITY OR FUNCTIONALITY?

- The first part of the work investigated the effect of the L-PBF process parameters (PPs) on the **density** and on the **Transformation Temperatures** that dictate the NiTi functional behavior
- 34 different combinations of PPs were investigated
- The aim was to avoid PPs leading to low density and/or cracking



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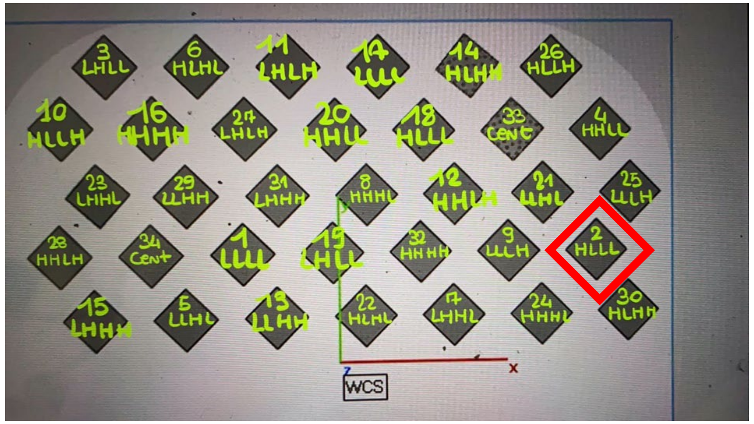

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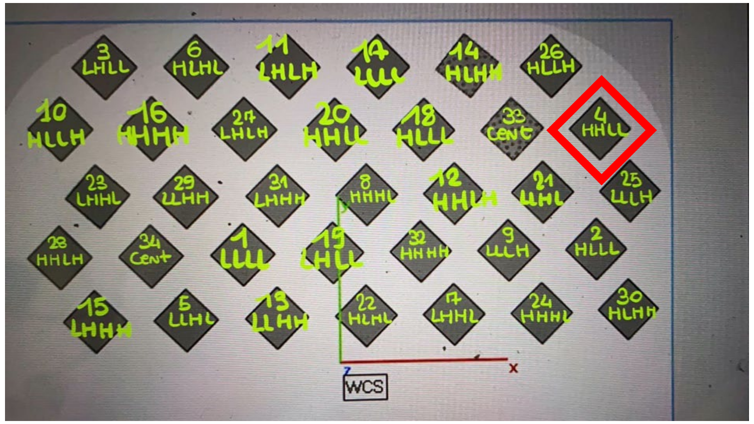

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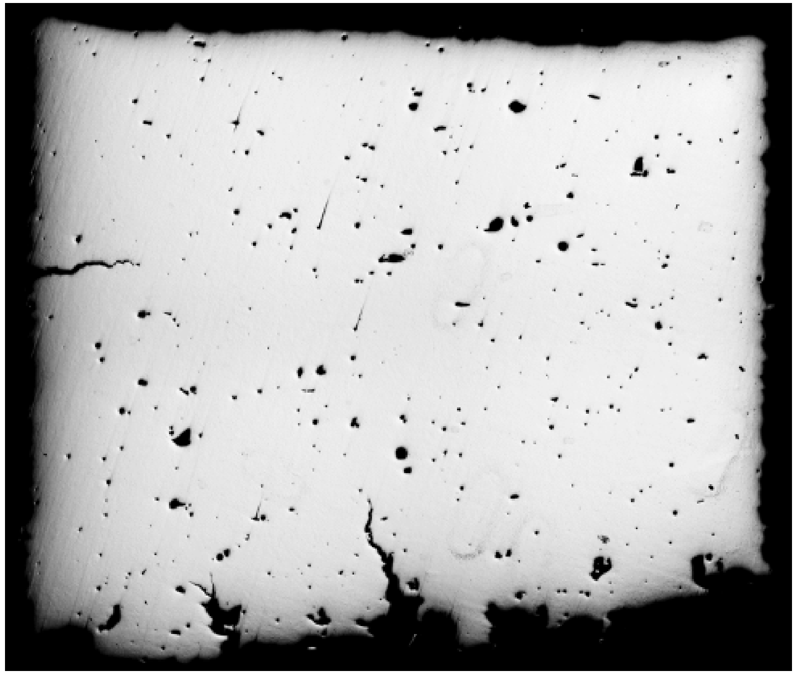

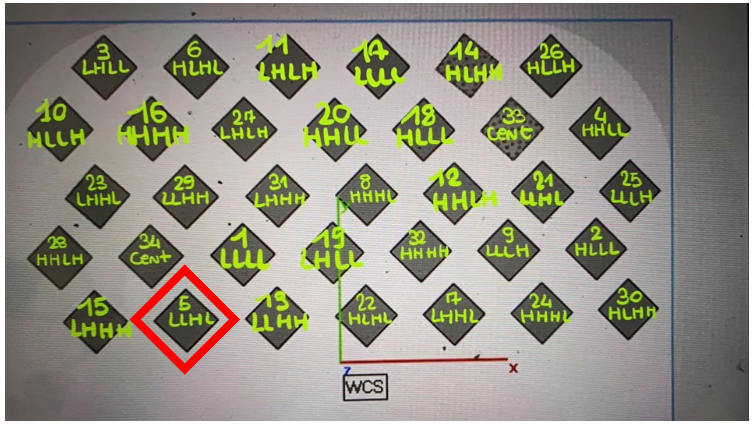

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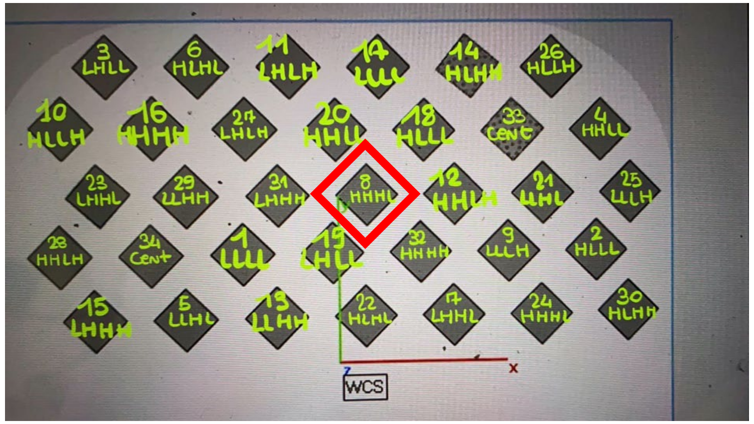
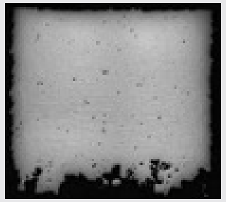
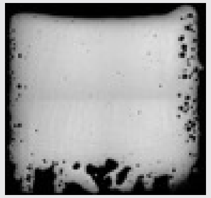
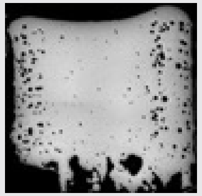
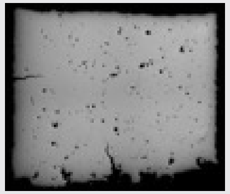
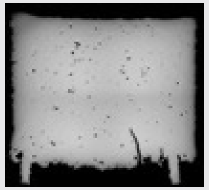
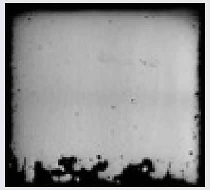
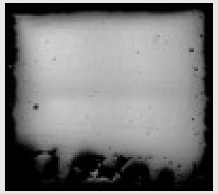
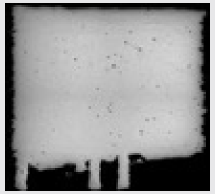
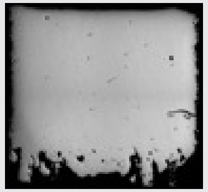
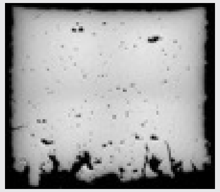
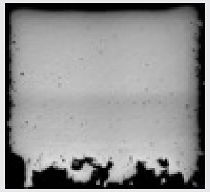


Process parameters

1: LLLL	2:HLLL	3:LHLL	4:HHLL	5:LLHL	6:HLHL	7:LHHL	8:HHHL
Density [%] 99,36	Density [%] 97,45	Density [%] Failed	Density [%] 94,78	Density [%] 99,21	Density [%] 99,37	Density [%] 99,22	Density [%] 99,35
		X					

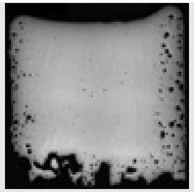
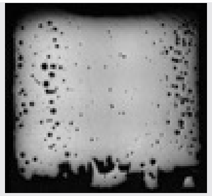
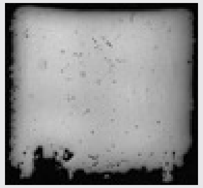
9:LLLH	10:HLLH	11:LHLH	12:HHLH	13:LLHH	14:HLHH	15:LHHH	16:HHHH
Density [%] 99,68	Density [%] 99,63	Density [%] 99,41	Density [%] 99,39	Density [%] 96,97	Density [%] 99,25	Density [%] 99	Density [%] 99,52
							

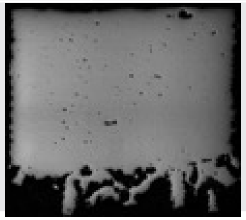
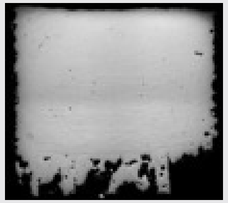
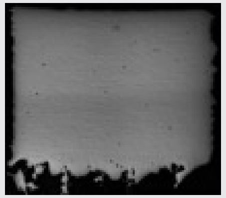
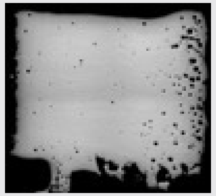
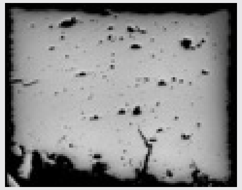
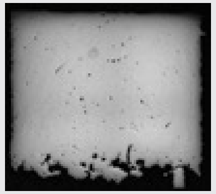
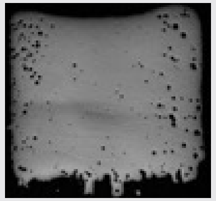
33:Cent	
Density[%]	99,52
	

Additive 4 Biomedical

SC 09/A3 CoCo AM Additive Manufacturing

PROCESS PARAMETERS

17:LLLL	18:HLLL	19:LHLL	20;HHLL	21:LLHL	22:HLHL	23:LHHL	24:HHHL
Density [%] 99,37	Density [%] 97,45	Density [%] Failed	Density [%] 96,22	Density [%] 99,34	Density [%] 99,62	Density [%] 99,7	Density [%] 99,3
		X					

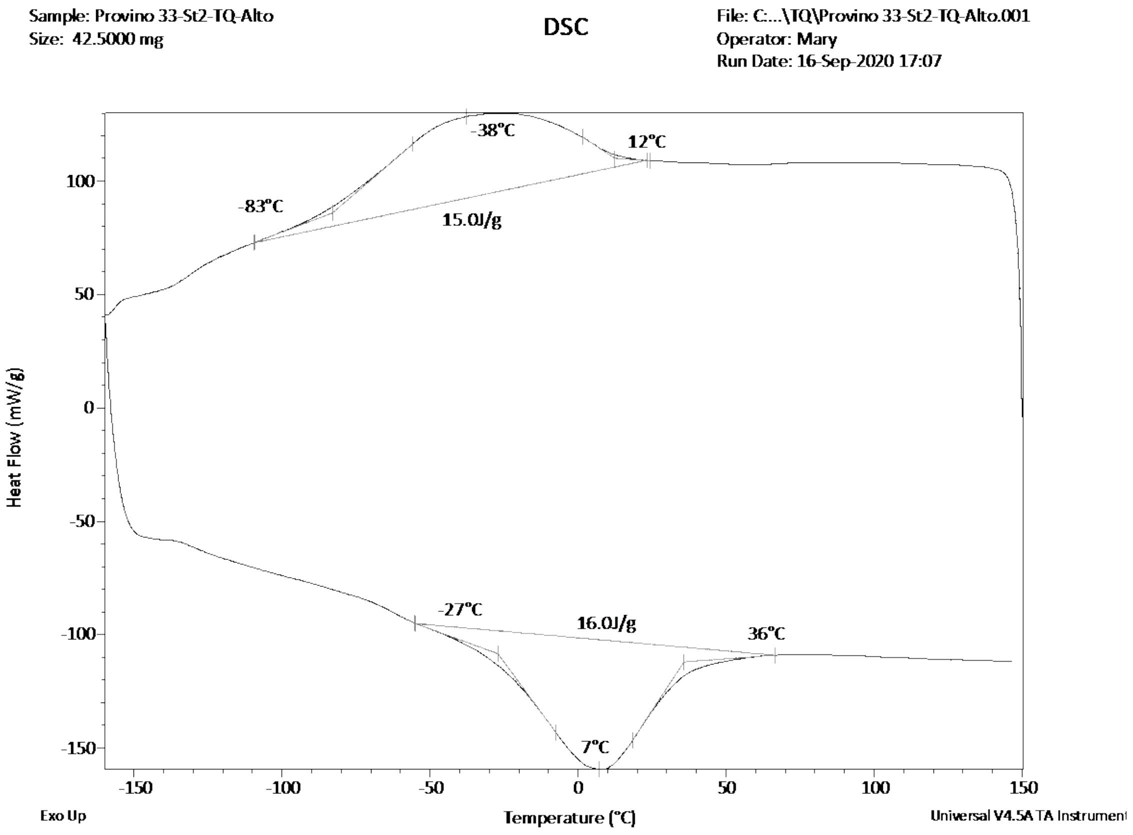
25:LLLH	26:HLLH	27:LHLH	28:HHLH	29:LLHH	30:HLHH	31:LHHH	32:HHHH
Density[%] 99,01	Density [%] 99,13	Density [%] 99,65	Density [%] 97,55	Density [%] 97,94	Density [%] 99,33	Density [%] 99,18	Density [%] 97,65
							

34:Cent	
Density[%]	99,53
	

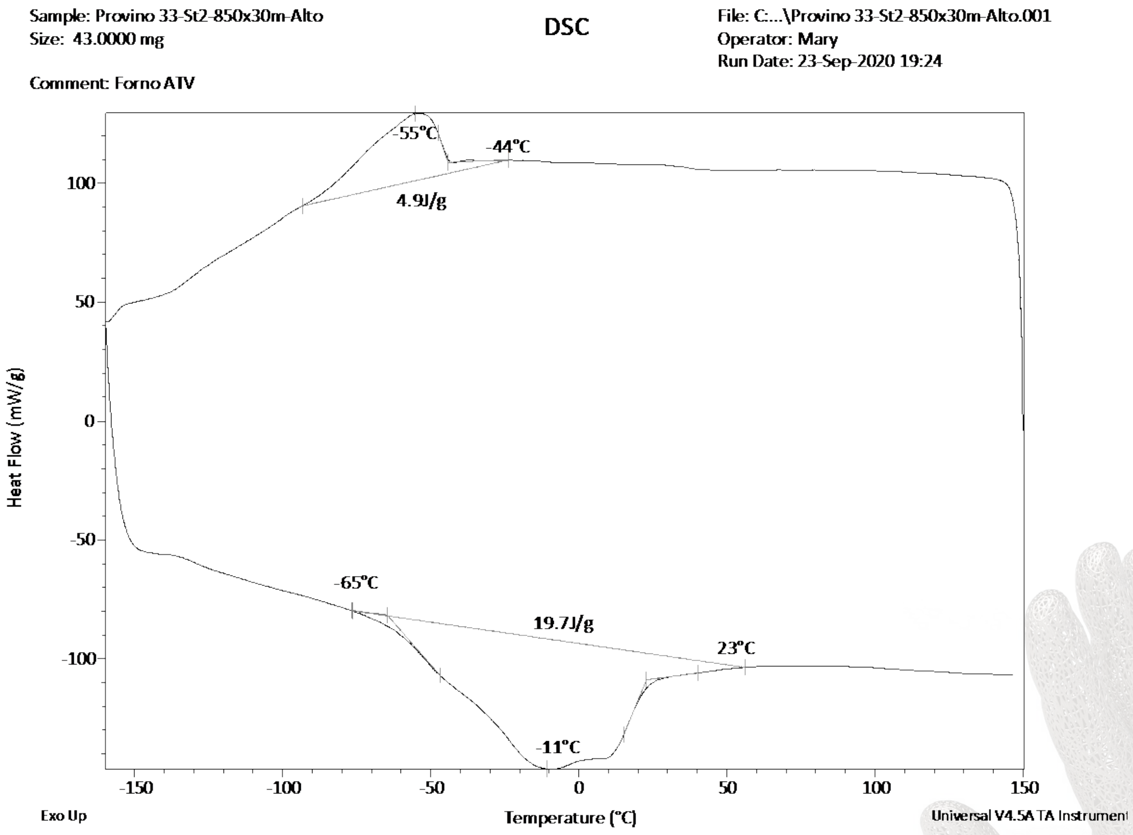
ADDITIVE 4 BIOMEDICAL

Full density or functionality?

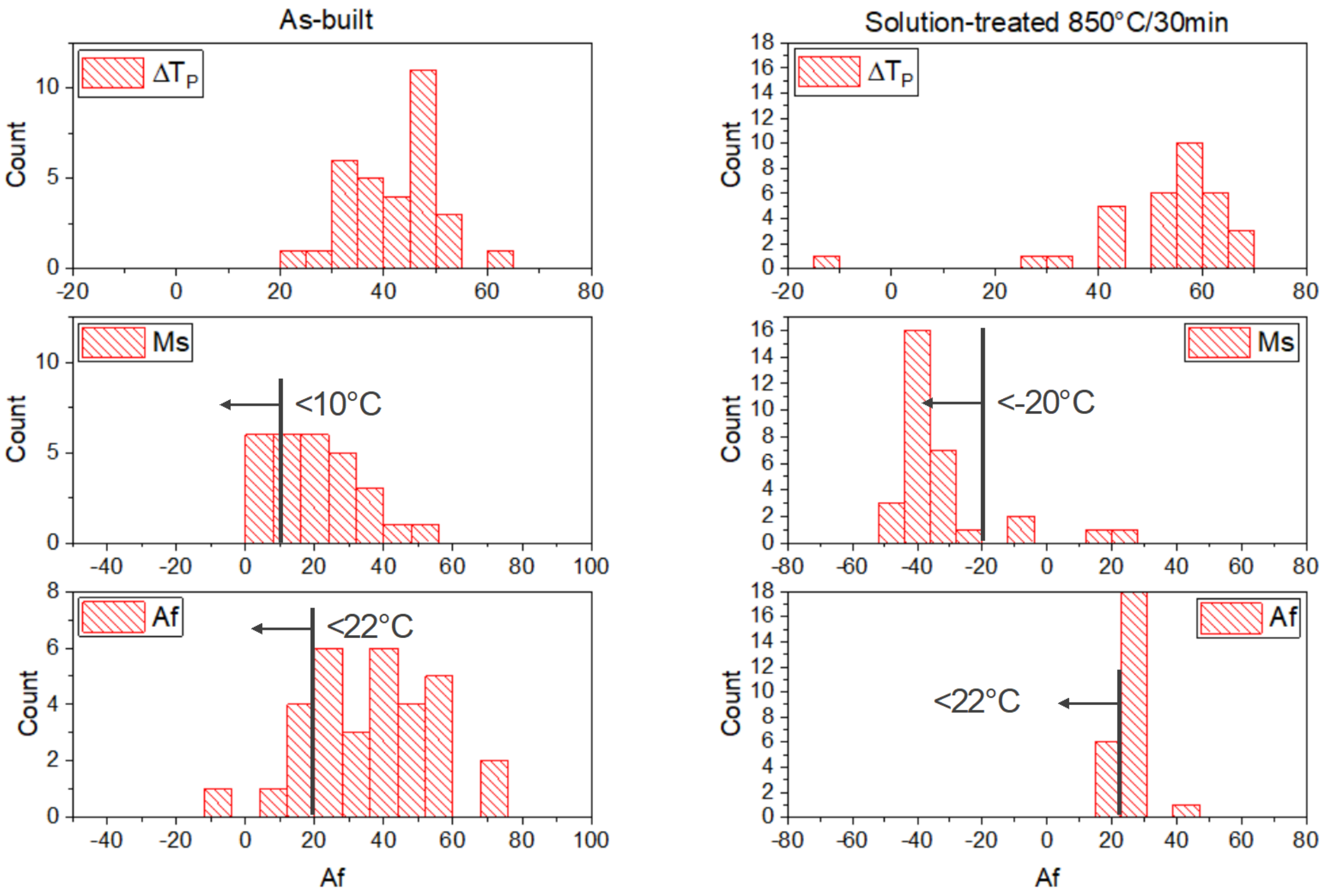
P33 – As-Built



P33 – 850°C/30min



PROCESS PARAMETERS AND TRANSFORMATION TEMPERATURES



The objective is to obtain superelastic response at Room Temperature

- **Ms**: Martensite Start Temperature
- **As**: Austenite Start Temperature
- **Mf**: Martensite Finish Temperature
- **Af**: Austenite Finish Temperature

Process parameters and Transformation Temperatures

Camp.ALTO	AB	As	Ap	Af	Mf	Mp	Ms	ΔTp	ΔHA	Note
Provino 1-ST2	√	−32	18	52	−82	-18	29	36	18,2	
Provino 2-ST2	√	−21	6	26	−76	-35	15	41	17,6	
Provino 3-ST2	√	−72	-39	−10		-61		22	11,3	
Provino 4-ST2	√	−2	26	44	−58	-23	24	49	19,9	
Provino 5-ST2	√	−74	-28	15		-73		45	14,4	
Provino 6-ST2	√	−48	-8	16	-91	-56	5	48	16,8	
Provino 7-ST2	√	−71	-20	19		-65		45	11,9	
Provino 8-ST2	√	−40	-3	18	-88	-57	1	54	18,4	
Provino 9-ST2	√	−21	16	56	-86	-14	32	30	21,8	
Provino 10-ST2	√	−3	31	55	−45	-2	36	33	18,2	2 M peak
Provino 11-ST2	√	2	44	69	-39	1	45	43	18,6	
Provino 12-ST2	√	−15	16	42	-68	-21	18	37	19	
Provino 13-ST2	√	−80	-37			-		#VALUE!	11,8	No M Peak
Provino 14-ST2	√	−33	11	32	-92	-34	16	45	20,1	
Provino 15-ST2	√	−66	-7	37		-39	14	32	18	
Provino 16-ST2	√	−7	23	51	-51	-12	26	35	17,3	
Provino 33-ST2	√	-27	7	36	-83	-38	12	45	16	

Camp.ALTO	AB	As	Ap	Af	Mf	Mp	Ms	ΔTp	ΔHA	Note
Provino 17-ST2	√	−17	12	53	-51	-18	26	30	16,4	
Provino 18-ST2	√	−29	1	29	-86	-38	10	39	15,7	
Provino 19-ST2										
Provino 20-ST2	√	−19	5	21	-86	-55	5	60	10,9	Double M Peak
Provino 21-ST2	√	−74	-29	22		-75	3	46	12,3	
Provino 22-ST2	√	−43	-6	24	-94	-55	9	49	15,8	
Provino 23-ST2	√	−32	1	36	-87	-28	20	29	15,9	
Provino 24-ST2	√	−40	-7	20		-56	3	49	17,5	
Provino 25-ST2	√	−32	15	58		-15	32	30	19,1	
Provino 26-ST2	√	−51	-6	24	-87	-54	11	48	18,3	
Provino 27-ST2	√	10	49	75	-25	12	52	37	22	
Provino 28-ST2	√	−78	29	50		-21	29	50	18,6	Elongated Peaks
Provino 29-ST2	√	-75	-32	7		-74		42	13,6	
Provino 30-ST2	√	-13	12	38	-72	-31	17	43	12,6	
Provino 31-ST2	√	-45	-2	36	-92	-35	18	33	17,4	
Provino 32-ST2	√	-73	-6	49		-58	2	52	10,9	
Provino 34-ST2	√	-19	12	35	-82	-35	18	47	15,2	

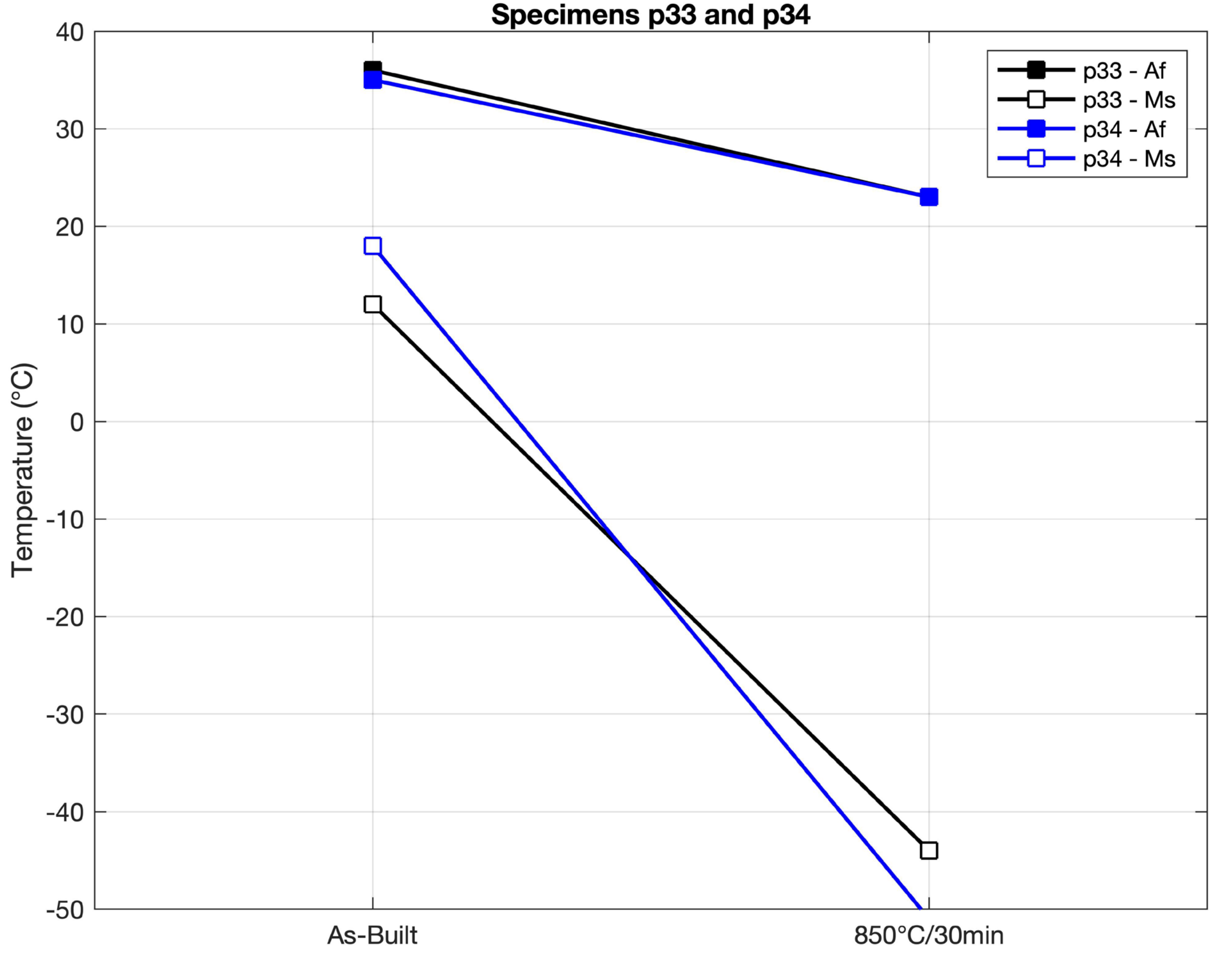
PROCESS PARAMETERS AND TRANSFORMATION TEMPERATURES

ADDITIVE 4 BIOMEDICAL

Camp.ALTO	Sol	As	Ap	Af	Mf	Mp	Ms	ΔTp	ΔHA	Note
Provino 1-ST2	√	–33	9	28	-87	-56	-7	65	19,6	Irregular M Peak
Provino 2-ST2	√	–40	-7	25	-94	-60	-42	53	19,7	
Provino 3-ST2	√	–75	5	27	-81	-60	-31	65	15,8	Low dH M Peak
Provino 4-ST2	√	–53	-18	25	-114	-76	-43	58	17,7	Small double peaks
Provino 5-ST2	√	–72	8	26	-94	-58	-37	66	18,5	
Provino 6-ST2	√	–48	-14	23	-95	-66	-44	52	20,6	
Provino 7-ST2	√	–58	10	27	-81	-53	-38	63	18,3	Irregular A peak
Provino 8-ST2	√	–57	11	25	-89	-51	-40	62	21	Small double peaks
Provino 9-ST2	√	–34	8	27	-83	-52	-33	60	22,4	Irregular M Peak
Provino 10-ST2	√	–19	11	24	-66	-45	14	56	19,9	Irregular M Peak
Provino 11-ST2	√	–36	-7	26	-97	-62	-45	55	18,4	
Provino 12-ST2	√	–43	-6	19	-80	-50	-40	44	20,5	Irregular M Peak
Provino 13-ST2	√	–10	15	26	-64	-44	-32	59	20,2	
Provino 14-ST2	√	–35	-2	23	-79	-57	-6	55	19,2	Irregular M Peak
Provino 15-ST2	√	–53	-11	21		-53	-34	42	18,9	
Provino 16-ST2	√	–21	6	24	-72	-48	-23	54	19,8	Irregular M Peak
Provino 33-ST2	√	-65	-11	23		-55	-44	44	19,7	Low dH M Peak

Camp.ALTO	Sol	As	Ap	Af	Mf	Mp	Ms	ΔTp	ΔHA	Note
Provino 17-ST2	√	–45	0	23	-93	-55	-39	55	19,7	
Provino 18-ST2	√	–61	9	23	-89	-51	-42	60	21	Irregular Peaks
Provino 19-ST2										
Provino 20-ST2	√	–64	7	29	-67	-49	-37	56	14,5	Low dH M Peak
Provino 21-ST2	√		7	22	-74	-49	-33	56	17,9	Irregular A Peak
Provino 22-ST2	√	–54	2	21	-90	-52	-39	54	19,5	
Provino 23-ST2	√	–37	-6	22	-89	-60	-42	54	18,6	
Provino 24-ST2	√	–68	-23	24	-64	-51	-43	28	14,9	Low dH M Peak
Provino 25-ST2	√	–37	3	25	-86	-53	-34	56	19	
Provino 26-ST2	√	–71	9	26	-79	-53	-42	62	17,5	Low dH M Peak
Provino 27-ST2	√	–3	26	43	-52	-16	23	42	21,8	
Provino 28-ST2	√	–65	-20	27		-52		32	15	Irregular Peaks
Provino 29-ST2	√		8	26	-79	-54	-33	62	18,9	
Provino 30-ST2	√	-25	4	23	-74	-54	-47	58	15,6	Irregular M Peak
Provino 31-ST2	√	-40	-8	22	-86	-52	-40	44	19,8	
Provino 32-ST2	√	-70	-13	27				-13	13,7	No M Peak
Provino 34-ST2	√	-31	0	23	-78	-54	-51	54	19,4	

FULL DENSITY OR FUNCTIONALITY?

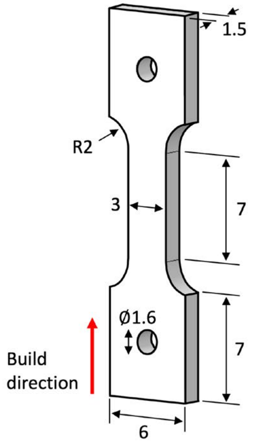


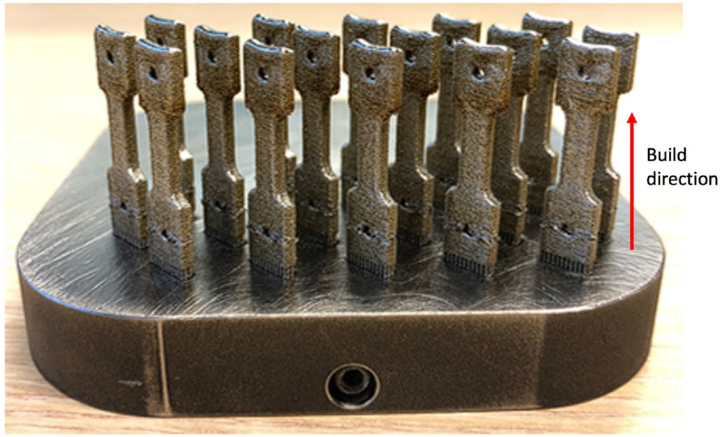
33:Cent	
Density[%]	99,52

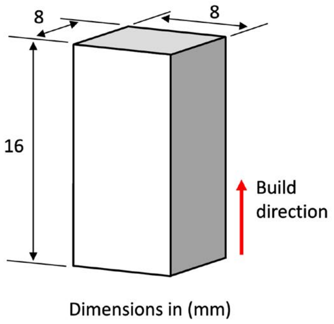
34:Cent	
Density[%]	99,53

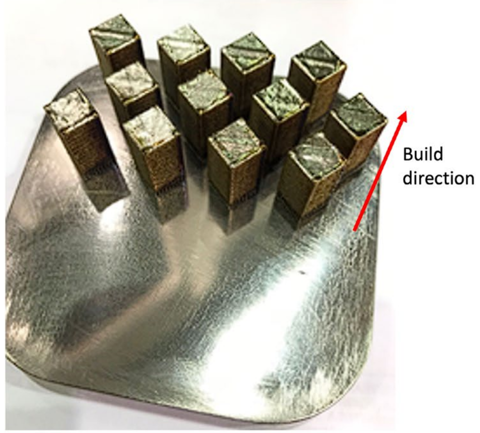
EXPERIMENTAL SET-UP

- **Tensile** dog-bone micro specimens were manufactured in two different orientations:
  - **Vertical**
  - **Horizontal**
- Solid **compressive** specimens were also manufactured



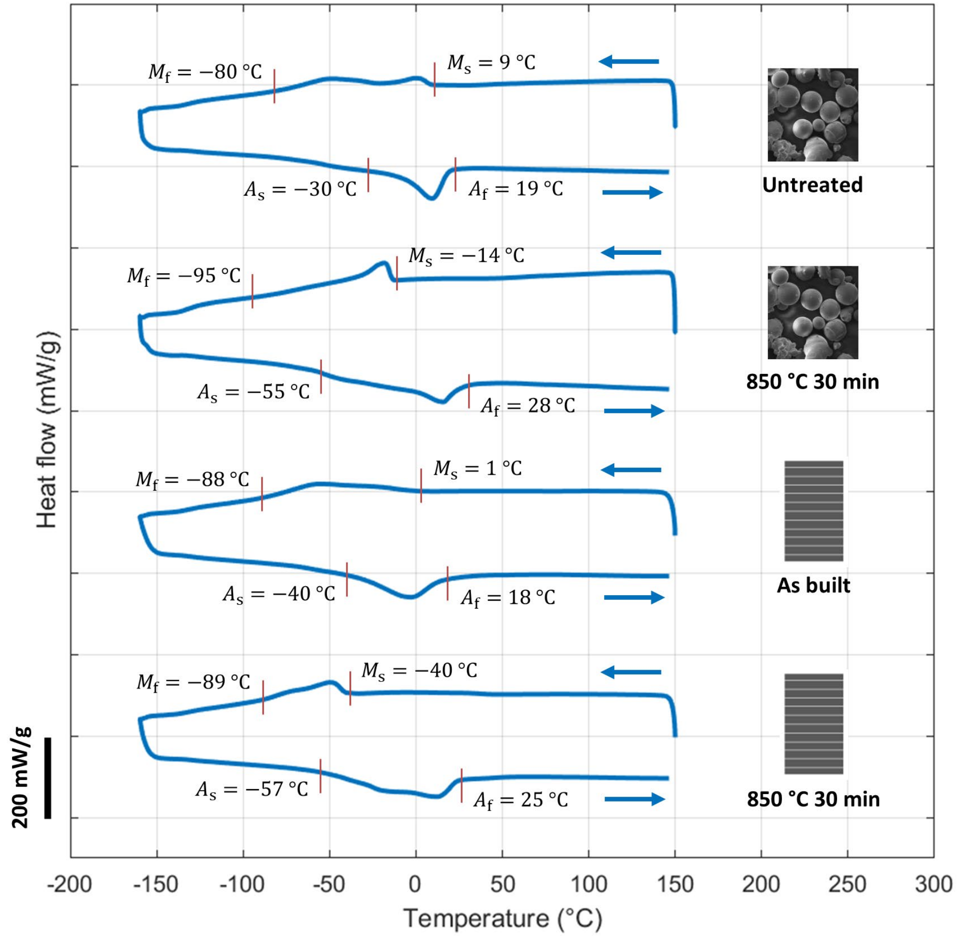






Carlucci, G., et al. "Building Orientation and Heat Treatments Effect on the Pseudoelastic Properties of NiTi Produced by LPBF." Shape Memory and Superelasticity (2022): 13.

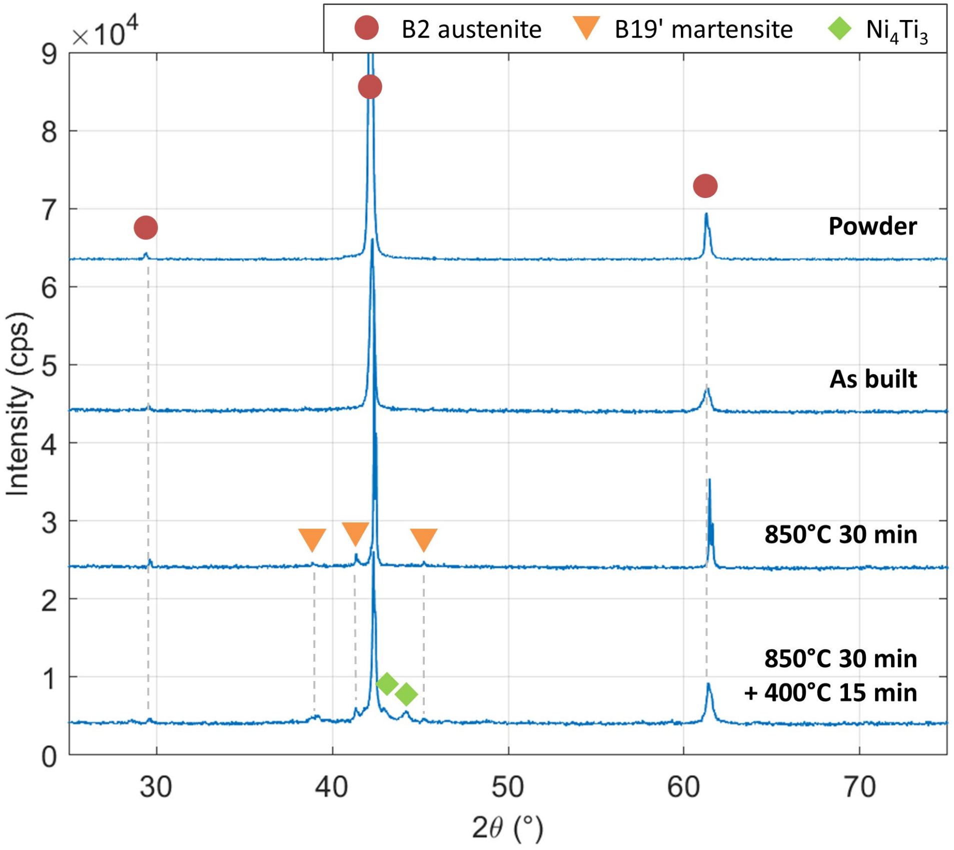
TRANSFORMATION TEMPERATURES

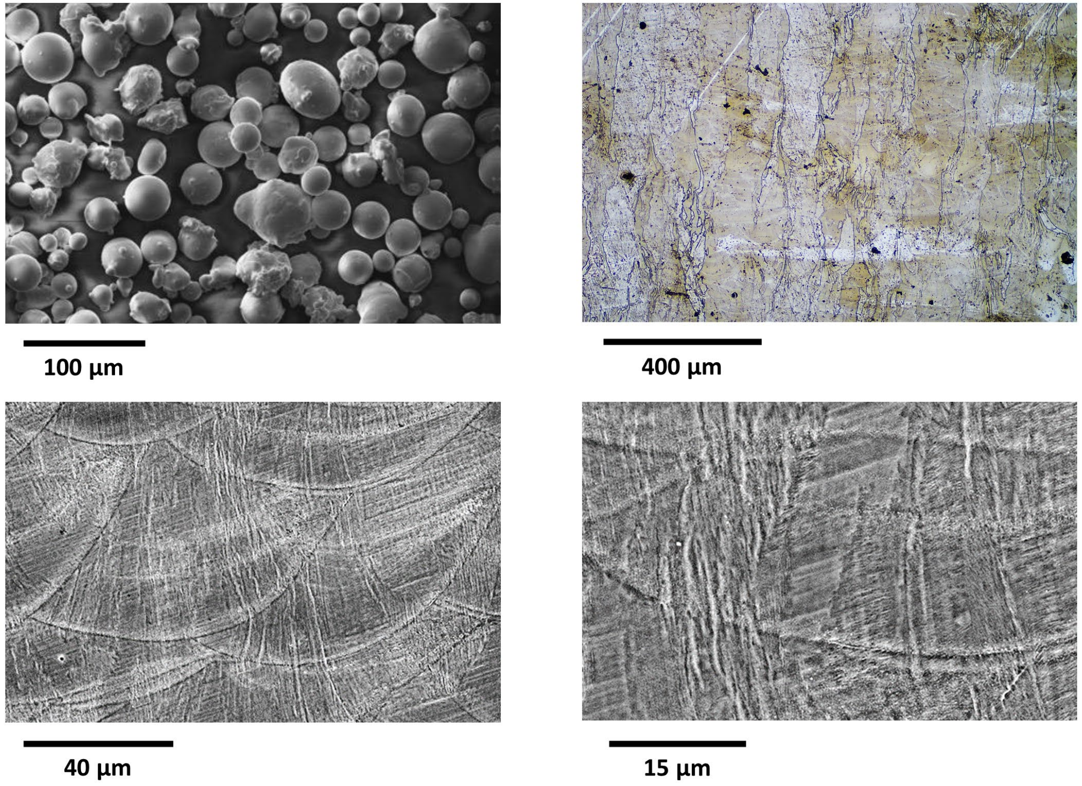


- **DSC analyses** were performed on gas-atomized powder and solution treated (850°C/1h) powder
- Additional DSC analyses were performed also on two compressive specimens to verify the Transformation Temperatures

Carlucci, G., et al. "Building Orientation and Heat Treatments Effect on the Pseudoelastic Properties of NiTi Produced by LPBF." Shape Memory and Superelasticity (2022): 13.

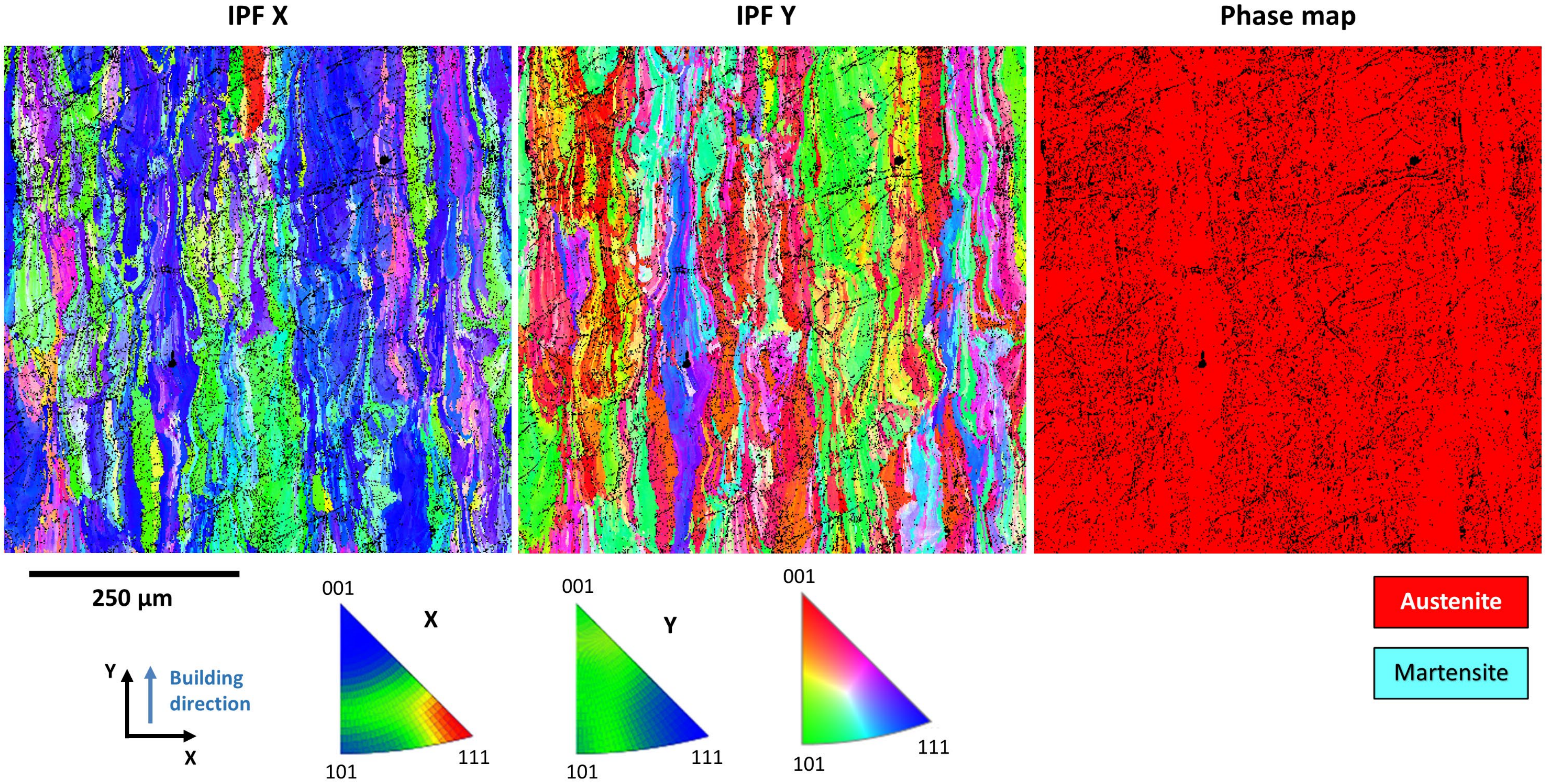
MICROSTRUCTURAL ANALYSIS





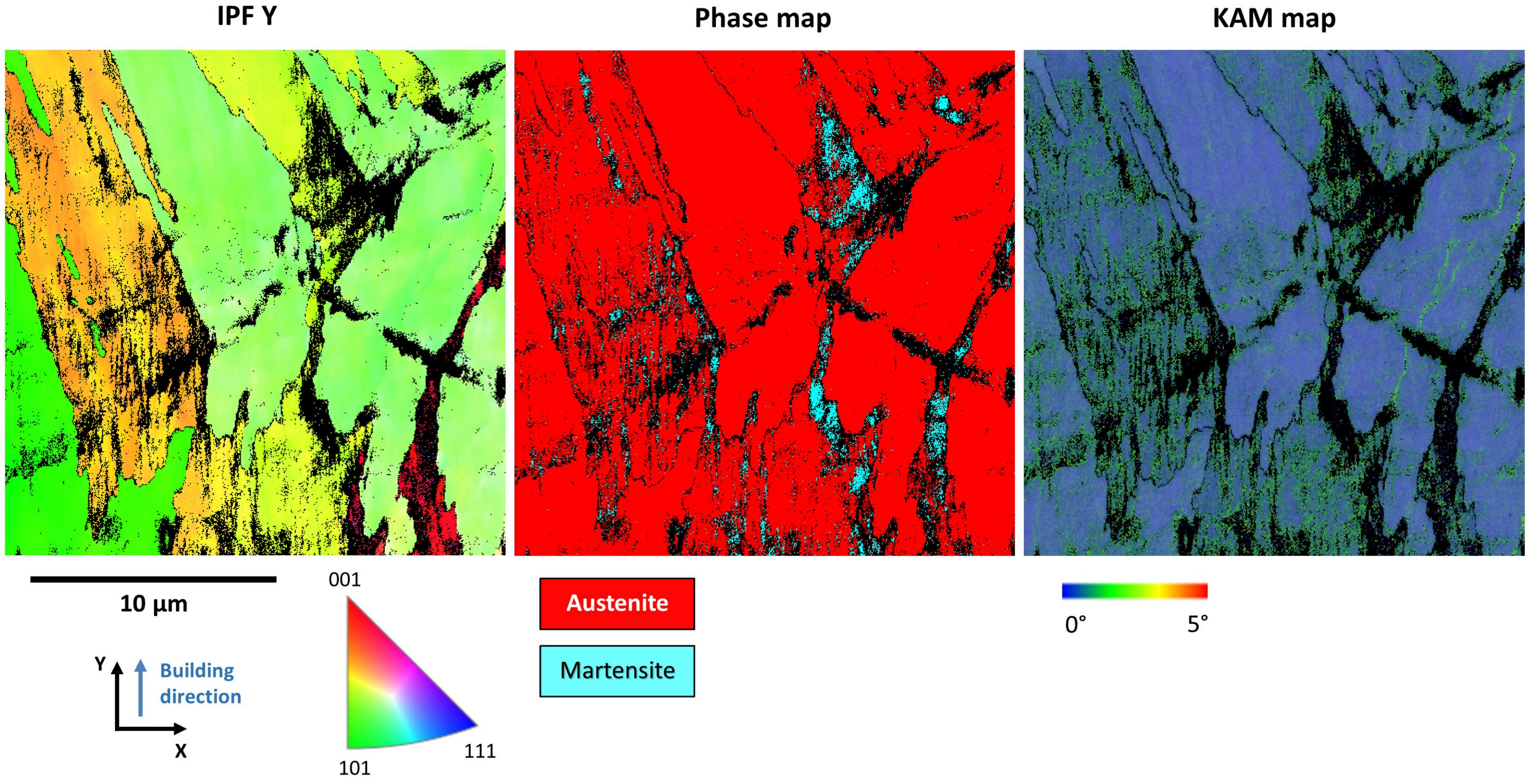
Carlucci, G., et al. "Building Orientation and Heat Treatments Effect on the Pseudoelastic Properties of NiTi Produced by LPBF." Shape Memory and Superelasticity (2022): 1-13.

EBSD – Low resolution



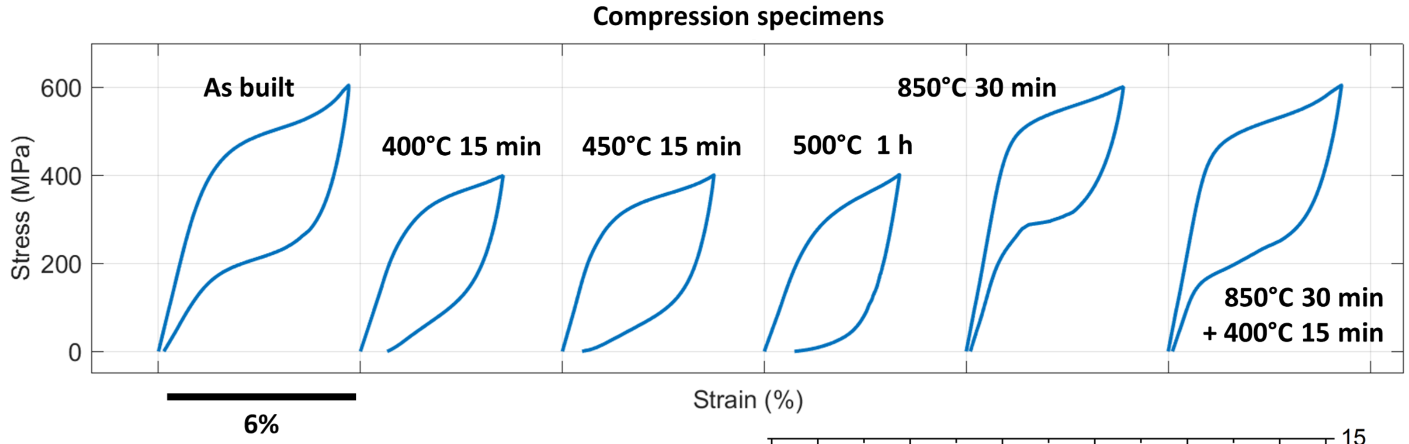
Carlucci, G., et al. "Building Orientation and Heat Treatments Effect on the Pseudoelastic Properties of NiTi Produced by LPBF." Shape Memory and Superelasticity (2022): 13.

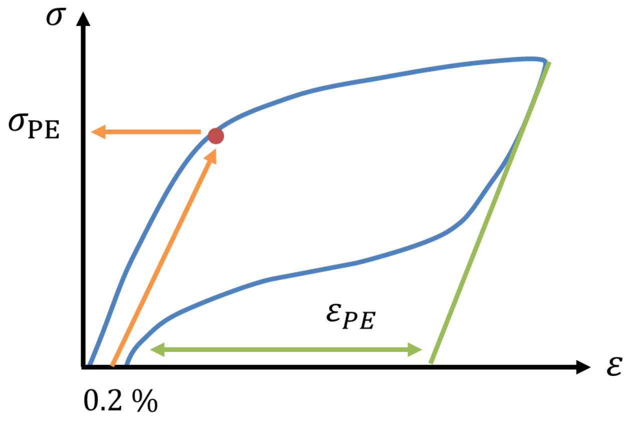
EBSD – High resolution

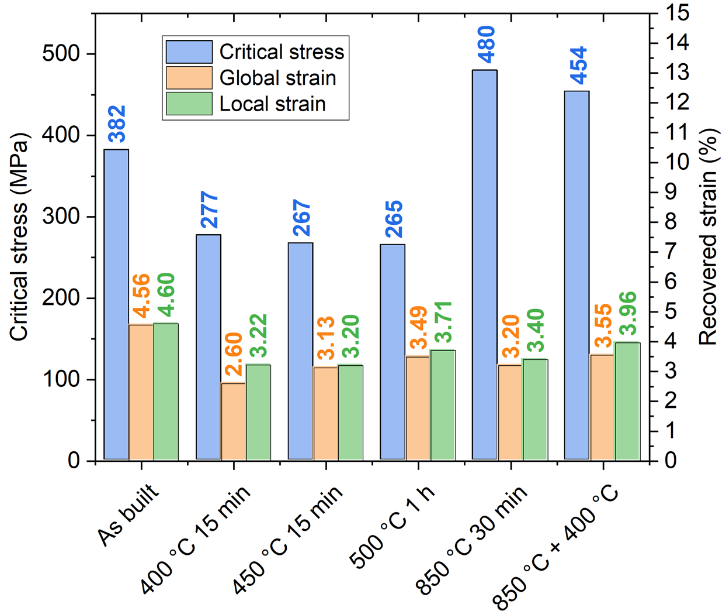


Carlucci, G., et al. "Building Orientation and Heat Treatments Effect on the Pseudoelastic Properties of NiTi Produced by LPBF." Shape Memory and Superelasticity (2022): 1-13.

Superelasticity in compression

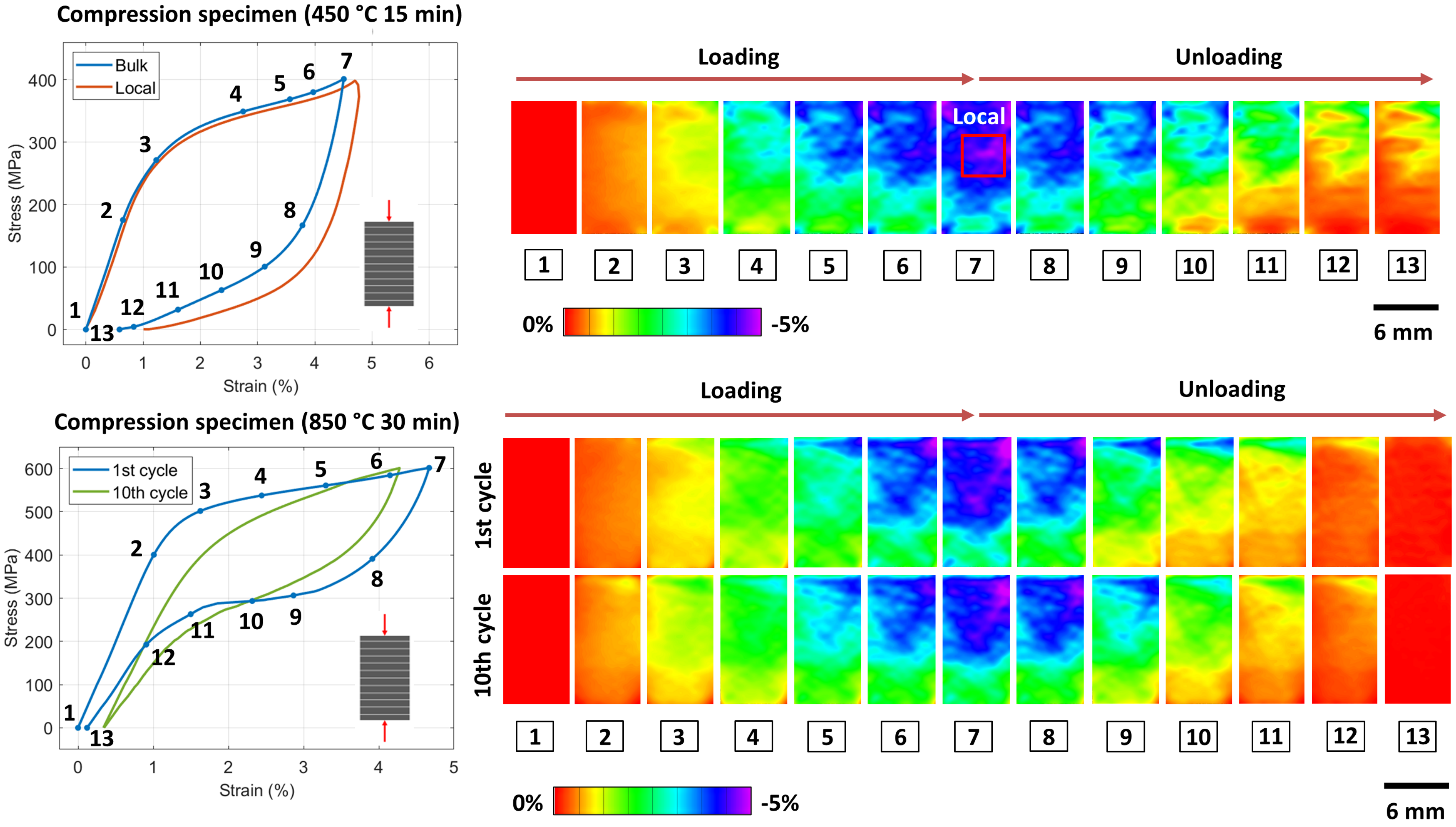






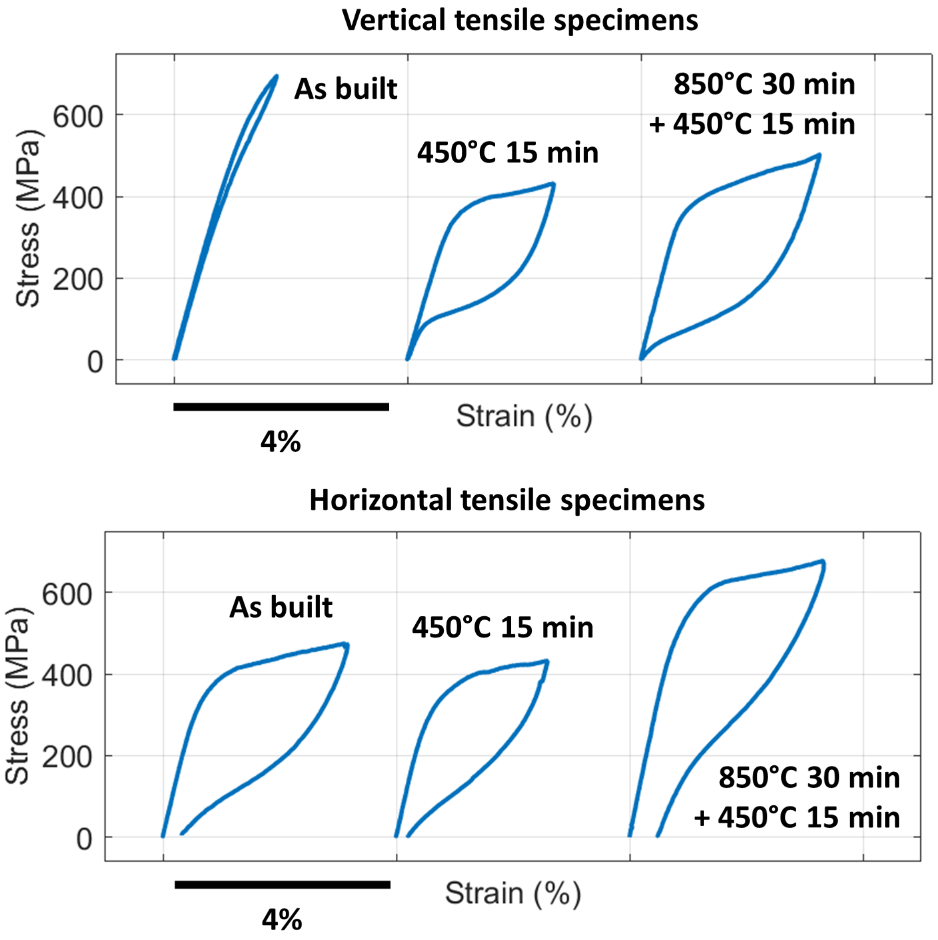
Carlucci, G., et al. "Building Orientation and Heat Treatments Effect on the Pseudoelastic Properties of NiTi Produced by LPBF." Shape Memory and Superelasticity (2022): 13.

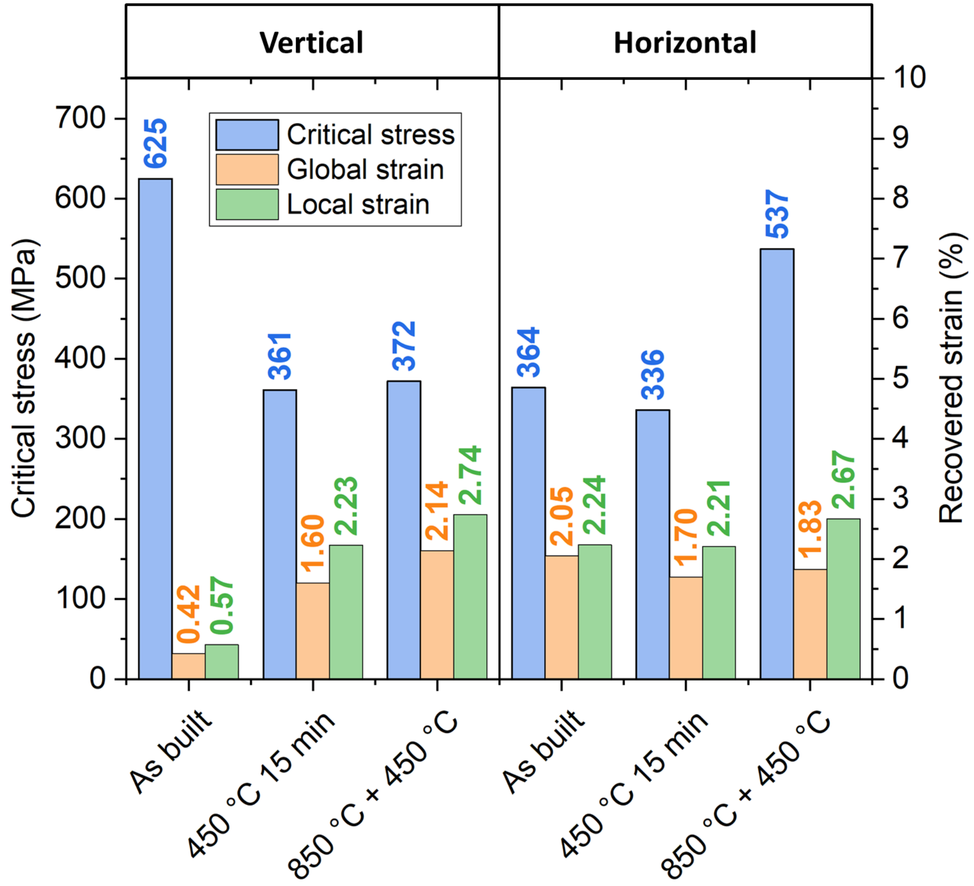
SUPERELASTICITY IN COMPRESSION



Carlucci, G., et al. "Building Orientation and Heat Treatments Effect on the Pseudoelastic Properties of NiTi Produced by LPBF." Shape Memory and Superelasticity (2022): 1-13.

TENSILE BEHAVIOR: VERTICAL vs HORIZONTAL





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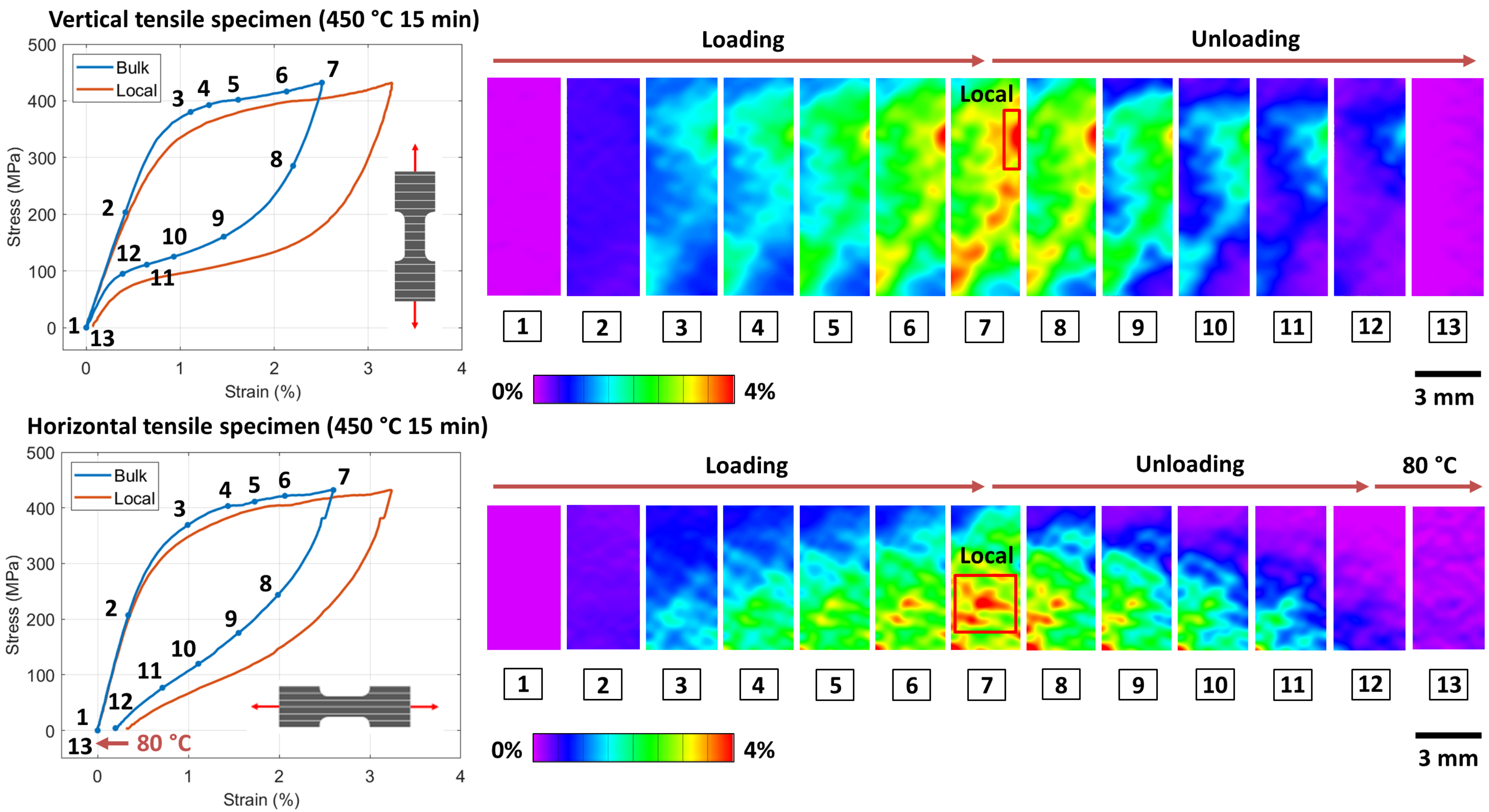
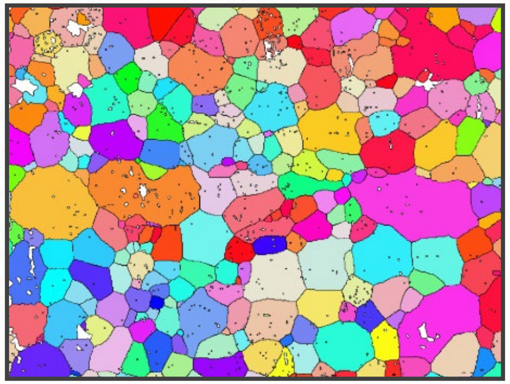
TENSILE BEHAVIOR: VERTICAL vs HORIZONTAL



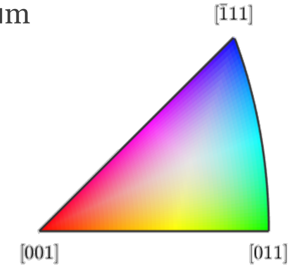
Carlucci, G., et al. "Building Orientation and Heat Treatments Effect on the Pseudoelastic Properties of NiTi Produced by LPBF." Shape Memory and Superelasticity (2022): 13.

COMPARISON BETWEEN THE MICROSTRUCTURES

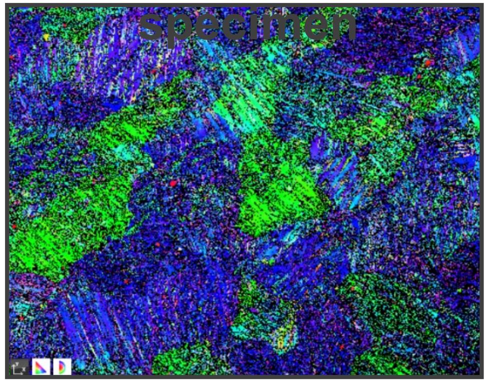
As cast



75 µm

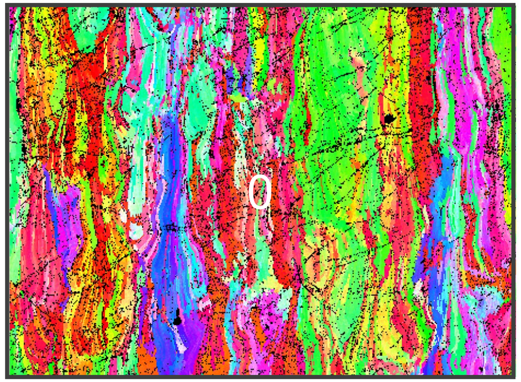


Multi-wires specimen

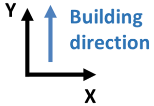


25 µm

L-PBF



250 µm



CONCLUSIONS

- NiTi superelastic behavior of AMed NiTi can be esily tailored to precise target operational temperatures
- A density of 99.5% was obtained according to the PPs study
- A maximum tensile transformation strain of 2.74% was obtained for the vertical specimens, while 2.64% for the horizontal ones
- Higher transformation strain (4.60%) was obtained in compression
- To compete with state-of-art stent manufacturing techniques:
  - Density has to be further required
  - Superelastic behavior of current NiTi stents can not be reached as the AMed NiTi is not texturized (texture favors higher transformation strains)
  - Surface roughness is still a problem, a surface treatment is required to remove asperities that are detrimental for fatigue performances