Metallurgy of ALM Nickel-based alloys: Which TTT diagrams ?

30-11-2022

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Agenda

01

SAFRAN Additive Manufacturing Campus

02

Case Study : Hastelloy® X

03

Conclusions and Outlook

SAMC, a key player for decarbonization in aeronautics

- 12 500 m² to closely connect
- AM design, research
- and production

THE DEVELOPMENT CENTER

40 design/production engineering staff specialized in AM

Calculation and simulation resources

THE PRODUCTION CENTER

75 operators and technicians qualified in AM

30 printers and heat treatment, chemical and finishing resources

Key milestones

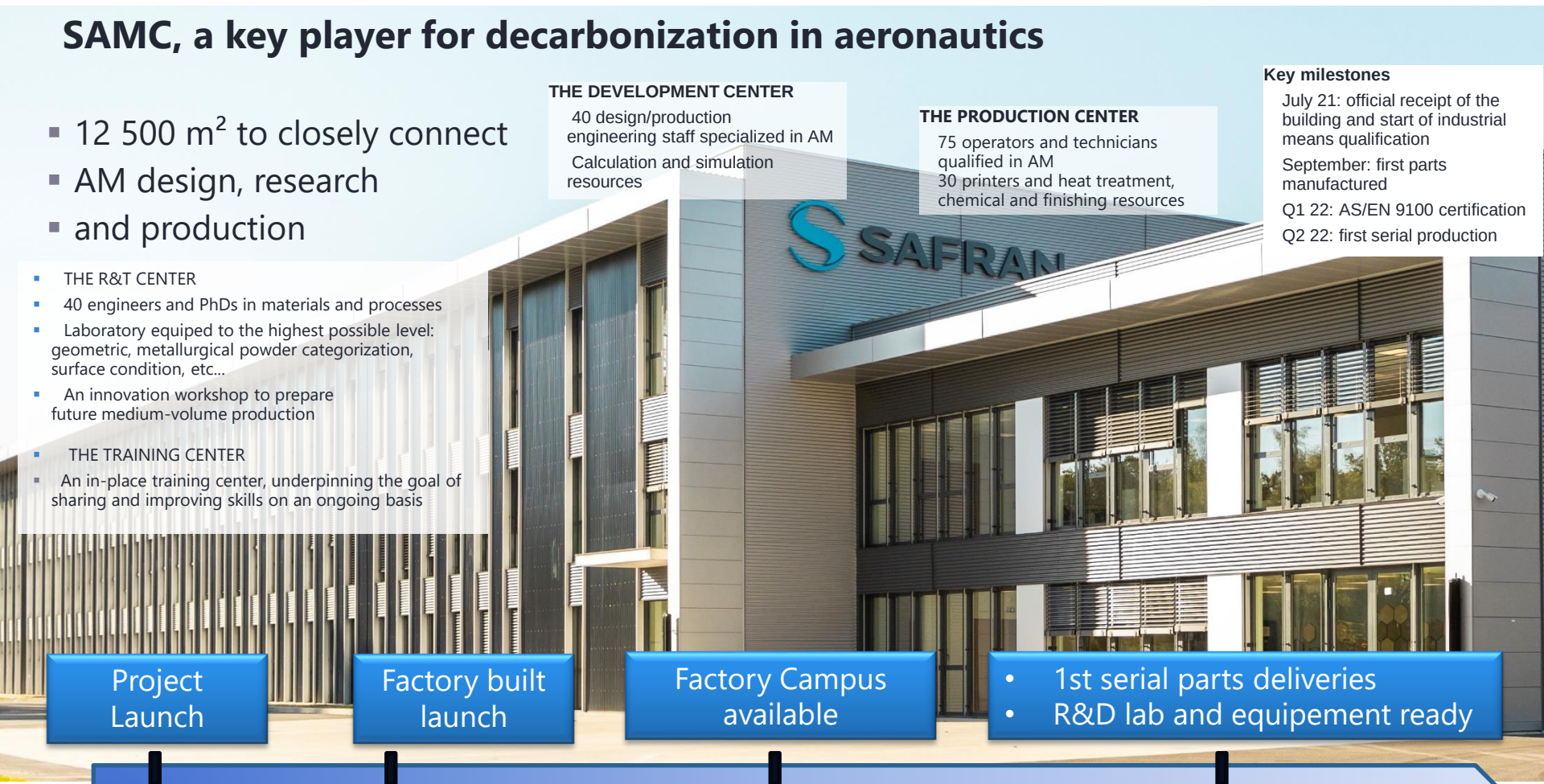
July 21: official receipt of the building and start of industrial means qualification

September: first parts manufactured

Q1 22: AS/EN 9100 certification

Q2 22: first serial production

- THE R&T CENTER
- 40 engineers and PhDs in materials and processes
- Laboratory equiped to the highest possible level: geometric, metallurgical powder categorization, surface condition, etc...
- An innovation workshop to prepare future medium-volume production
- THE TRAINING CENTER
- An in-place training center, underpinning the goal of sharing and improving skills on an ongoing basis



Project Launch	Factory built launch	Factory Campus available	• 1st serial parts deliveries • R&D lab and equipement ready
05/07/19	13/01/20	July/21	July 22

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Case Study: Hastelloy® X

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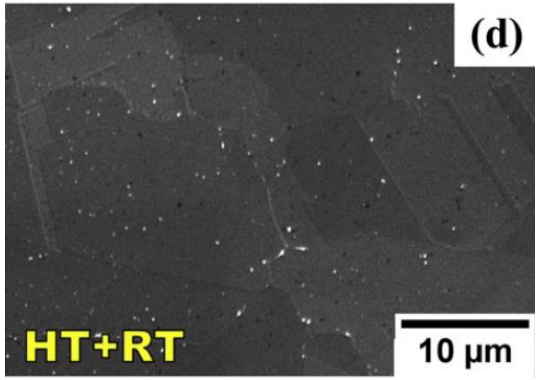
Case Study: Hastelloy® X - context

Chemical composition (wt%) : Nickel Base superalloy

	Ni	Fe	Cr	Mo	W	C	Co	Mn	Si
Min	Bal.	17,0	20,5	8,0	0,2		0,5		
Max		20,0	23,0	10,0	1,0	0,1	2,5	1,0	1,0

- Hastelloy® X microstructure:
  - ❖Solution hardening + carbides
  - ❖No gamma prime

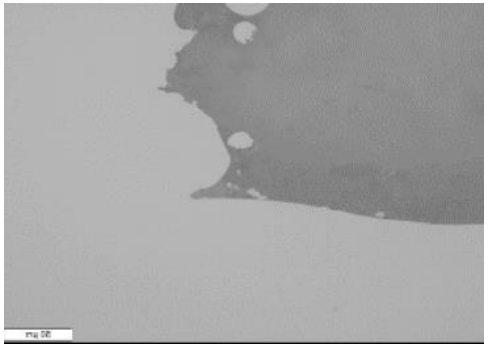
Annealing: 1177°C for 2h under Ar + air cooled



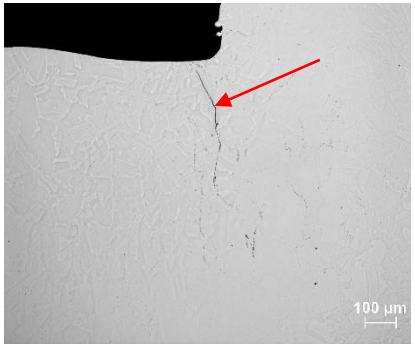
Sanchez-Mata et al., MSEA780 (2020) 139177

Case Study: Hastelloy® X - context

Before Heat Treatment (HT)



After Heat Treatment



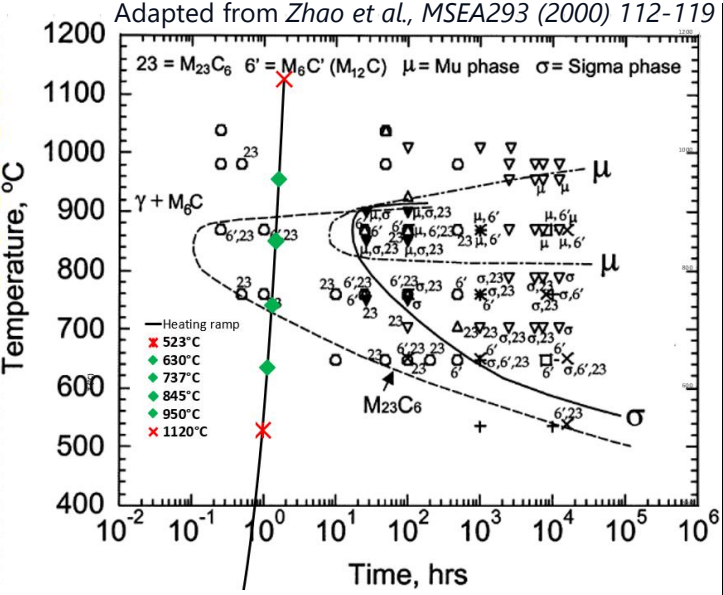
Building direction

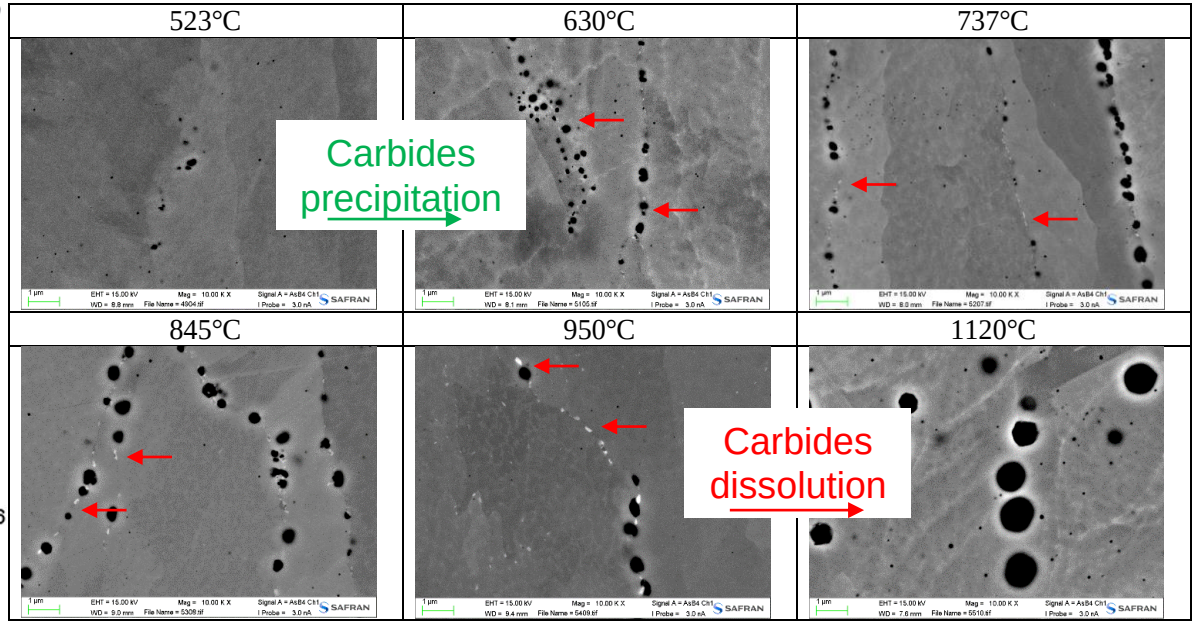
- Voids appear:
  - Well aligned
  - No clear link with microstructure (i.e. not at grain boundaries)
  - Some voids are connected (red arrow)

Case Study: Hastelloy® X - first trial

HT interrupted: heating ramp 10°C/min + water quench

Adapted from *Zhao et al., MSEA293 (2000) 112-119*





Necessity to Up-Date TTT diagram

Montero-Sistiaga works had the same conclusion
Montero-Sistiaga et al. Additive Manufacturing 31 (2020)

Case Study: Hastelloy® X - experimental procedure

- Small samples electro-machined in an as built bar, ~2x2x2mm to avoid thermal inertia
- Hot furnace stabilized, at least, 1h before sample introduction
  - Thermocouple in the furnace close to the crucible
  - Furnace stabilized in less than 5min after introduction
- Water quench in large water volume at room temperature
- Time-Temperature conditions:
  - Between 1 to 100h (need to fit in 6 month internship)
  - Between 600 to 1000°C based on literature data (mainly, Zhao et al., MSEA293 (2000) 112-119)
- SEM-EDS observation:
  - Sample are embedded + polished until 1µm
  - Carbon coating
  - EDS : 5kv / WD ~10mm

Case Study: Hastelloy® X - experimental procedure

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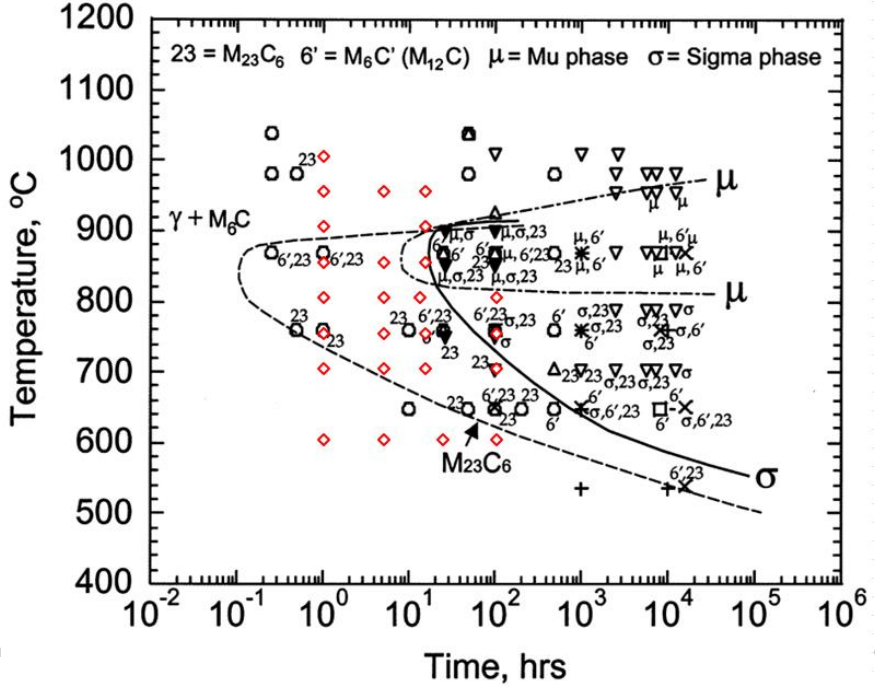
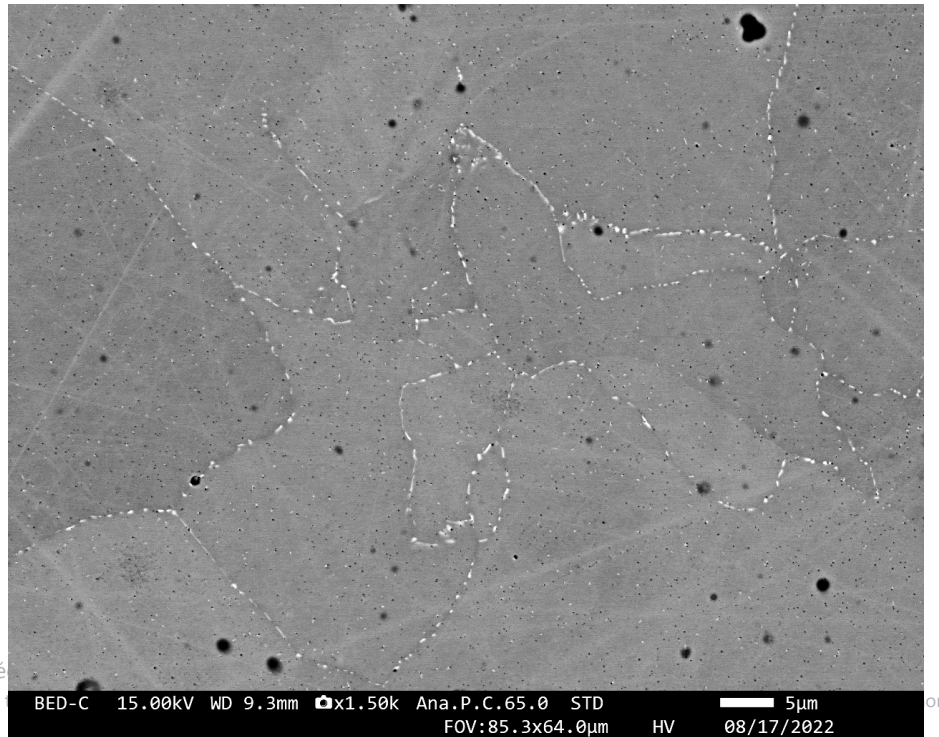


Adapted from *Zhao et al., MSEA293 (2000) 112-119*

Case Study: Hastelloy® X - Results

- 3 different phases identified:
  - First precipitation at grain boundaries

800°C/1h

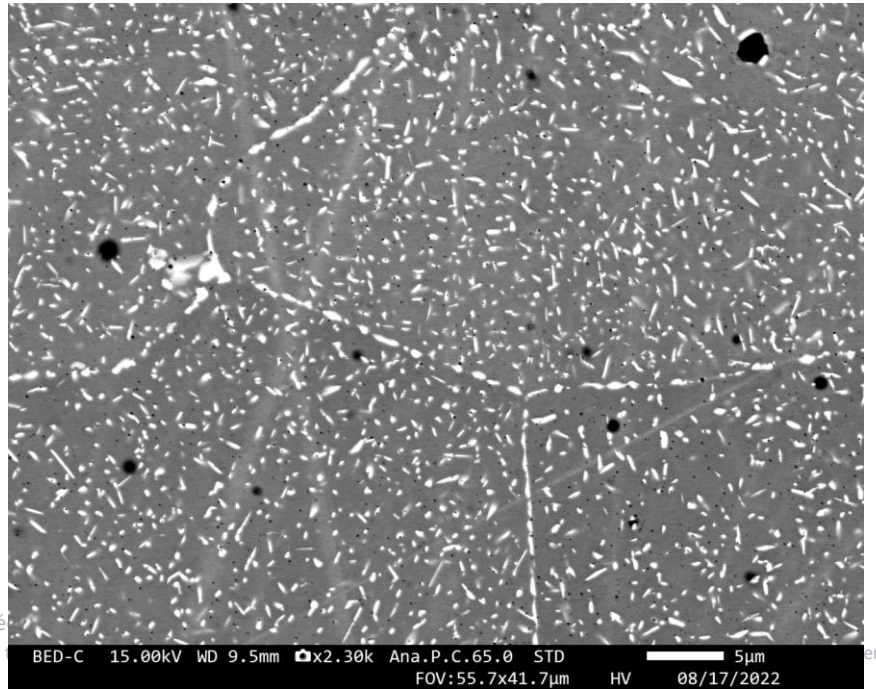


Case Study: Hastelloy® X - Results

- 3 different phases identified:
  - First precipitation at grain boundaries
  - Second phase inside of the grain with needle-shape

} Same chemical composition

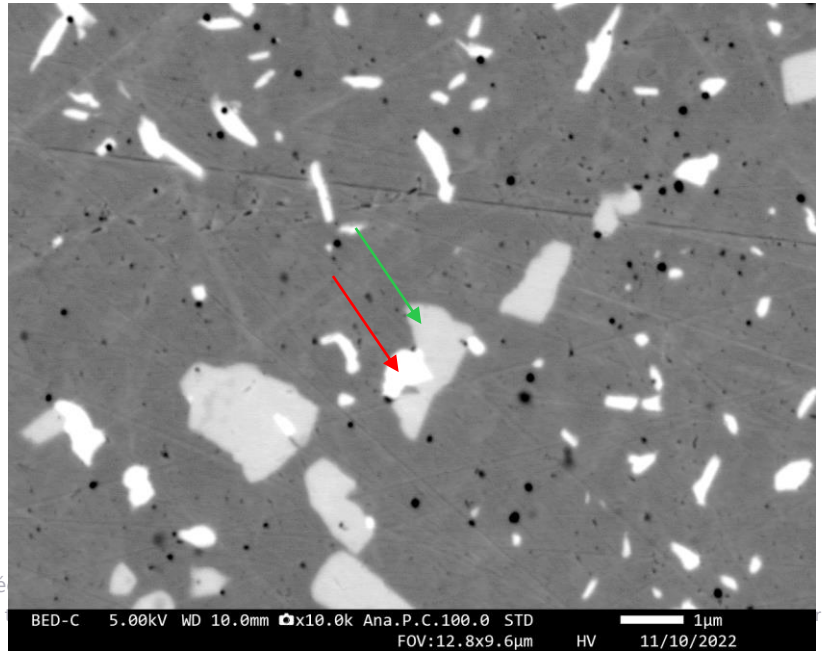
800°C / 13h



Case Study: Hastelloy® X - Results

- 3 different phases identified:
  - First precipitation at grain boundaries
  - Second phase inside of the grain with needle-shape
  - Last phase growth on the first phase under some conditions

800°C / 100h



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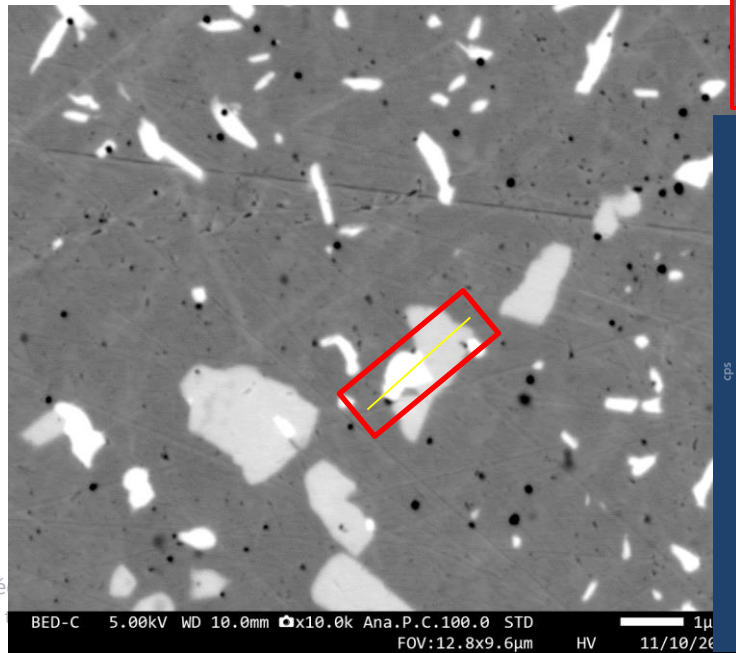
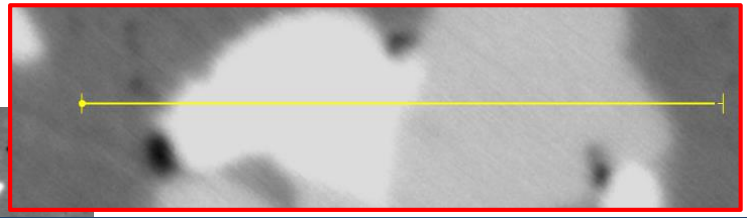
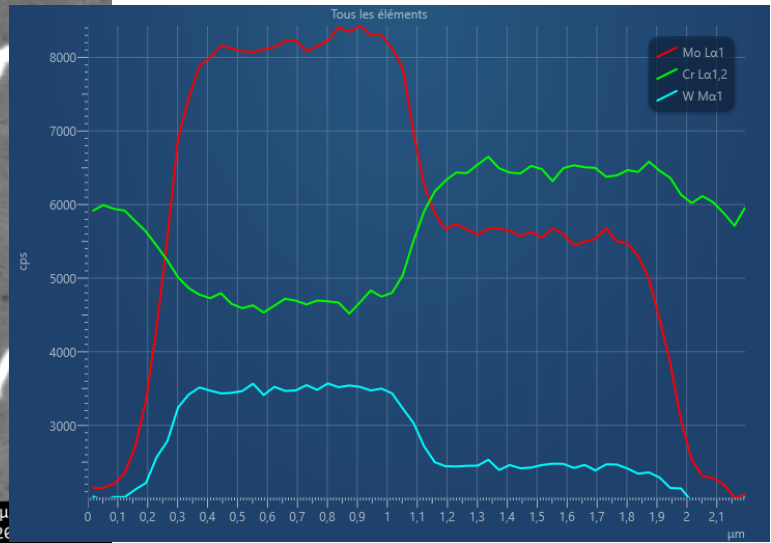
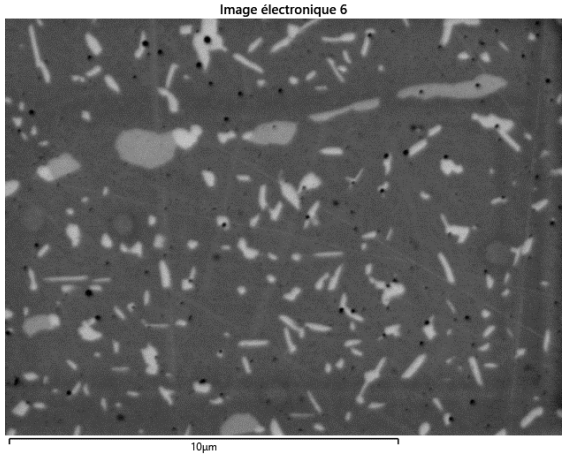






Case Study: Hastelloy® X - Results

750°C / 100h



- 3 different phases identified:
  - First precipitation at grain boundaries
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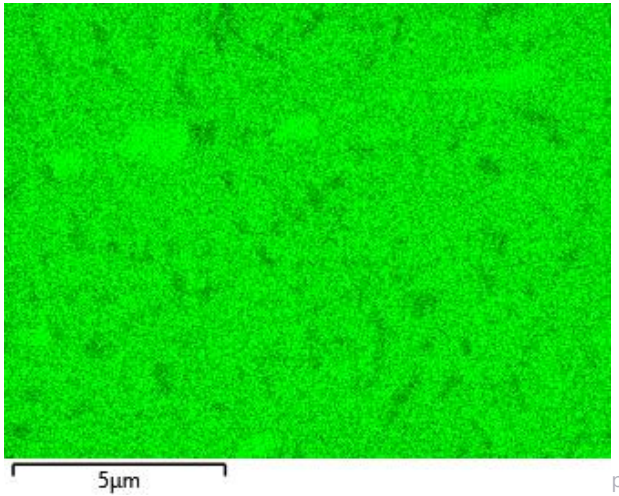
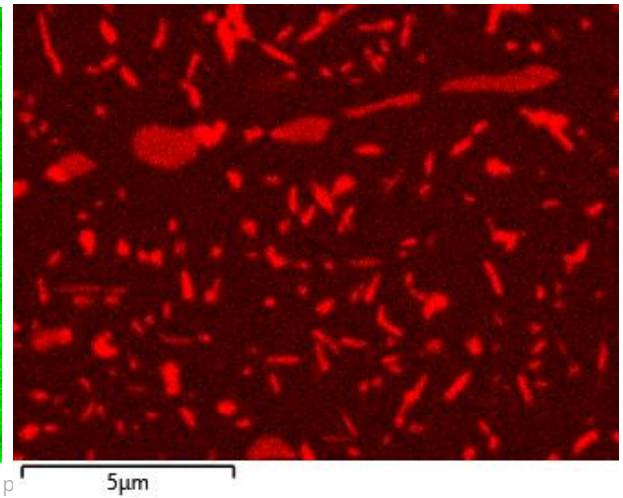
  - Last phase growth on the first phase under some conditions

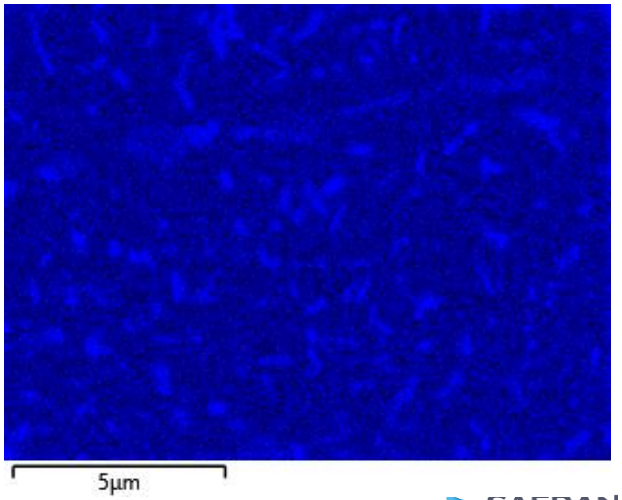
Cr



Mo



W



Case Study: Hastelloy® X - Results

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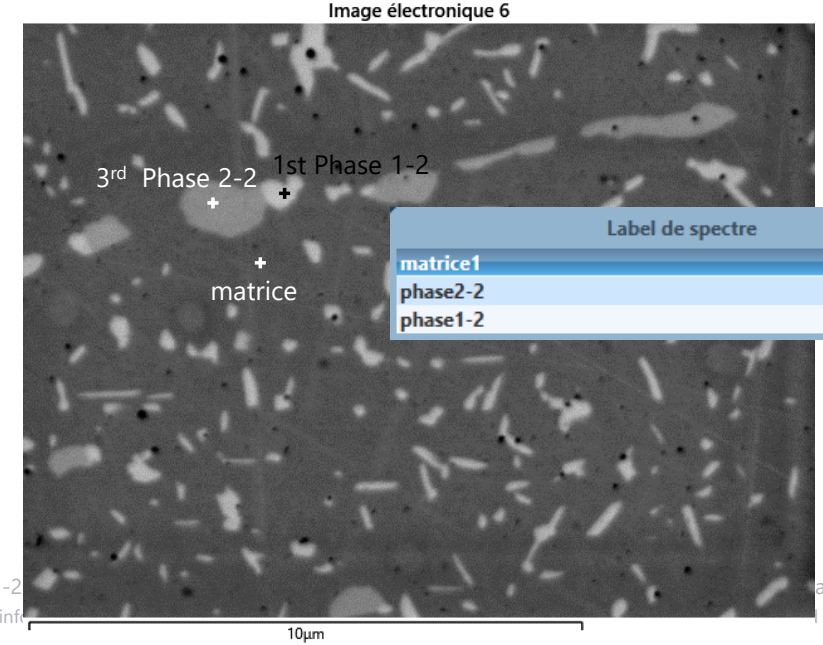


Wt%

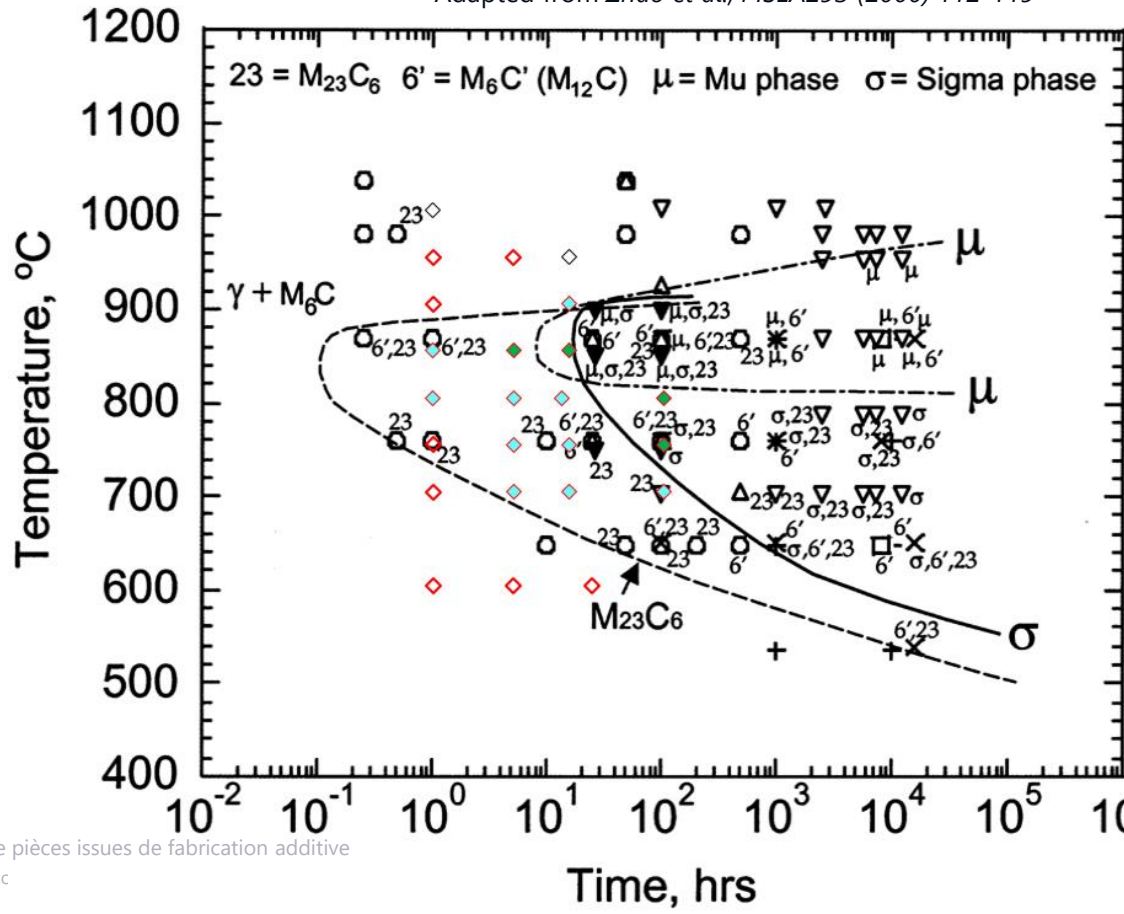
Label de spectre	Al	Ti	Cr	Fe	Co	Ni	Mo	W	Total
matrice1			18.37	19.69	1.54	53.78	5.53	1.11	100.00
phase2-2	0.26	0.00	28.31	14.68	1.92	25.25	26.88	2.70	100.00
phase1-2	0.27	0.00	12.83	13.38	1.94	22.61	43.76	5.21	100.00

Carbone numerically remove

Case Study: Hastelloy® X - Results

- First precipitation at grain boundaries
- First and Second phases
- All phases present
- No phase

Adapted from *Zhao et al., MSEA293 (2000) 112-119*

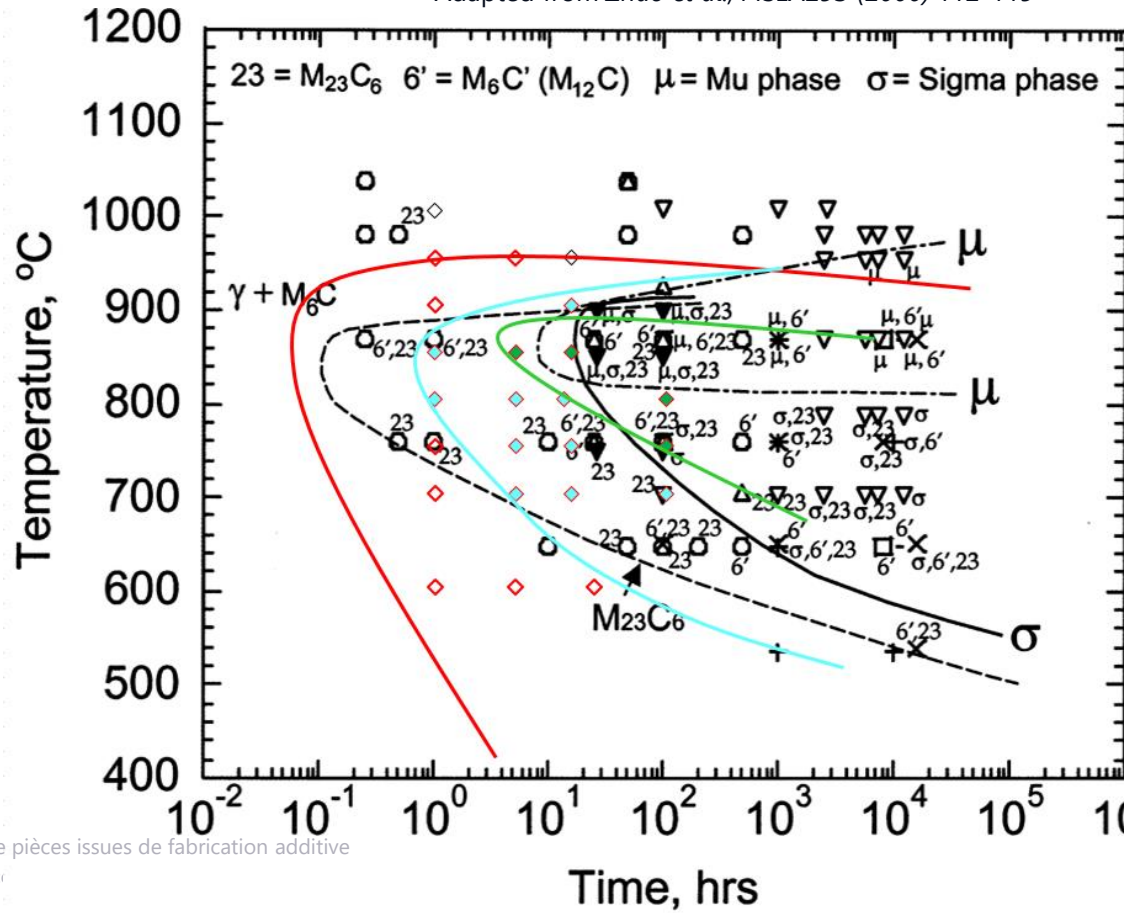


Case Study: Hastelloy® X - Results

- First precipitation at grain boundaries
- First and Second phases
- All phases present
- No phase

At that point, we cannot clearly identify the phases

Adapted from *Zhao et al., MSEA293 (2000) 112-119*



Conclusions and outlook

Conclusions and Outlook

- Hastelloy® X:
  - Need a careful identification of the phases:
    - EDS/EBSD, on-going
    - X-ray diffraction or TEM to be done if needed
  - Some point can be done after annealing treatment also
- Need to be extend to other alloys, e.g. Inconel® 718
- Comparison and calibration with numerical tools (e.g. Thermo-calc/DICTRA), on-going