Réactivité d'un superalliage à base de nickel de fabrication additive lors de traitements de compaction isostatique à chaud

Journées A3TS
Traitements et parachèvements de pièces issues de fabrication additive
30/11/2022

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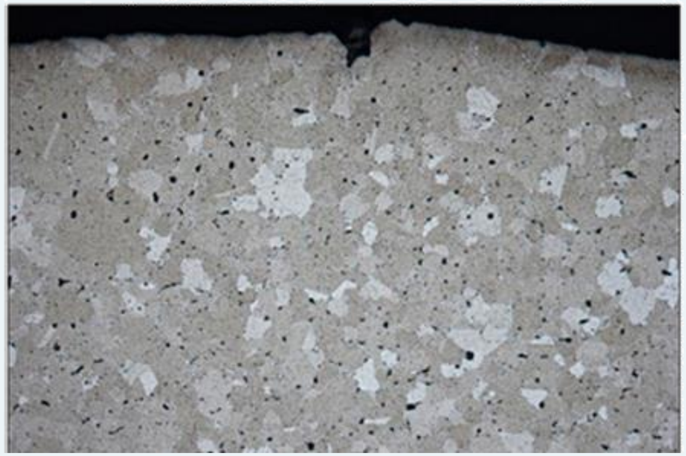
Introduction

Introduction: HIPPOME Project

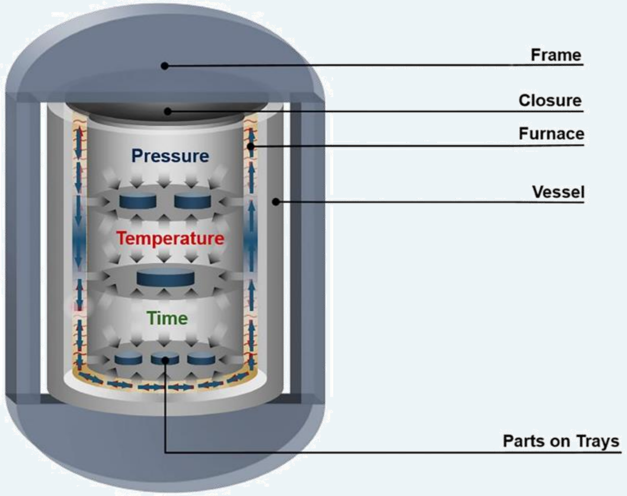


Example: Powder-metallurgy CM247LC with a high density of pores (black)

- Additive-manufacturing (AM) shaping process → defects such as porosity, cracks…

- Hot Isostatic Pressing (HIP) → close pores, consolidation of the material :
  - Very high temperature (>1000°C)
  - High total pressure (≥100 MPa)
  - Inert gas environment in a close system

- However, impurities traces found in HIP chambers (O_2, N_2, H_2O…)



Oxidation observed on post-HIP specimen surfaces

Introduction: HIPPOME Project

Short terms objectives

- Characterizing the oxide layer nature/structure post-HIP ;
- Improving our understanding of gaseous conditions during HIP = why do specimens oxidized ?
- Reproducing HIP oxidation at a laboratory scale

Controlling oxidation during HIP

Long terms objectives

Preventing any oxidation of the specimen during HIP
→ post-processing of net-shape parts

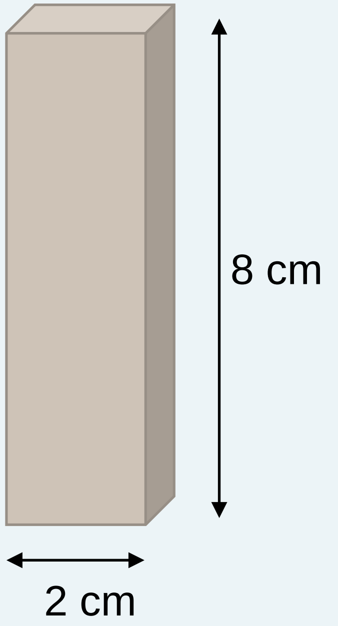
Plan of the presentation

1. As-built study material
2. Experimental strategy
3. Oxidation characterization

1. Material of study : As-built characterization

1. Material study – As-built characterization

Study material: a γ-γ' nickel-based superalloy as a AM rectangular bar



Wt% of Alloying Elements

Al	Ti	Cr
4.1	3.6	14.4

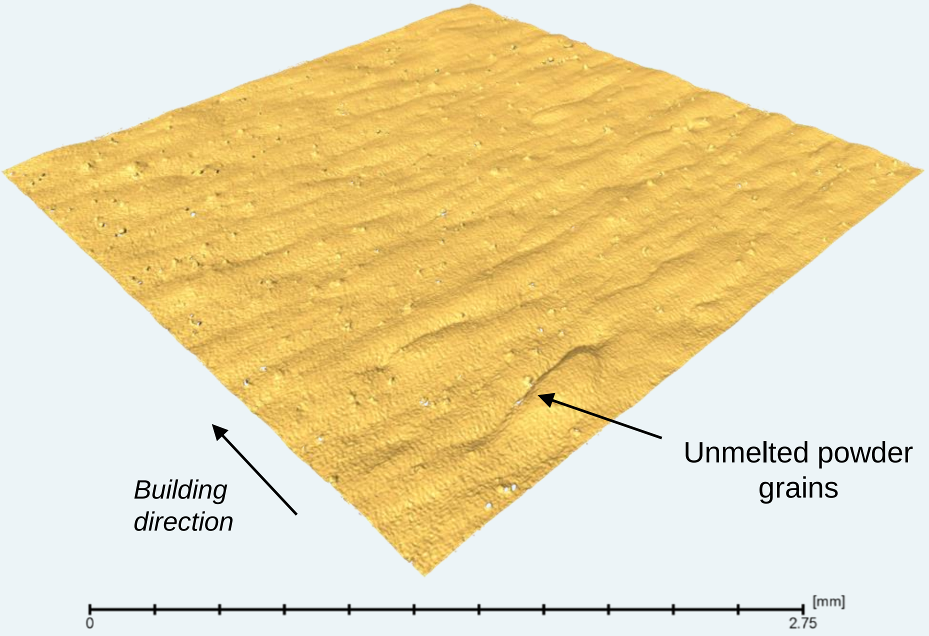
+ Co, Mo, Fe

As-built surface

P600 polished surface

Tomography

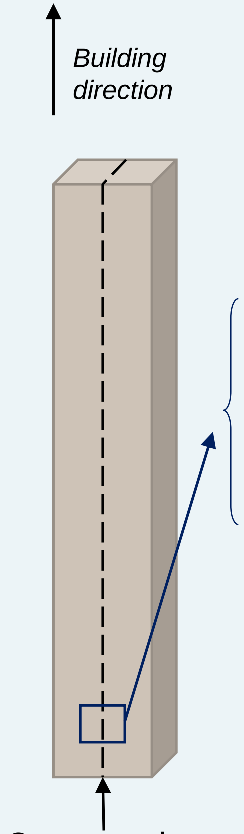
Extracted surface using adaptative subvoxel segmentation

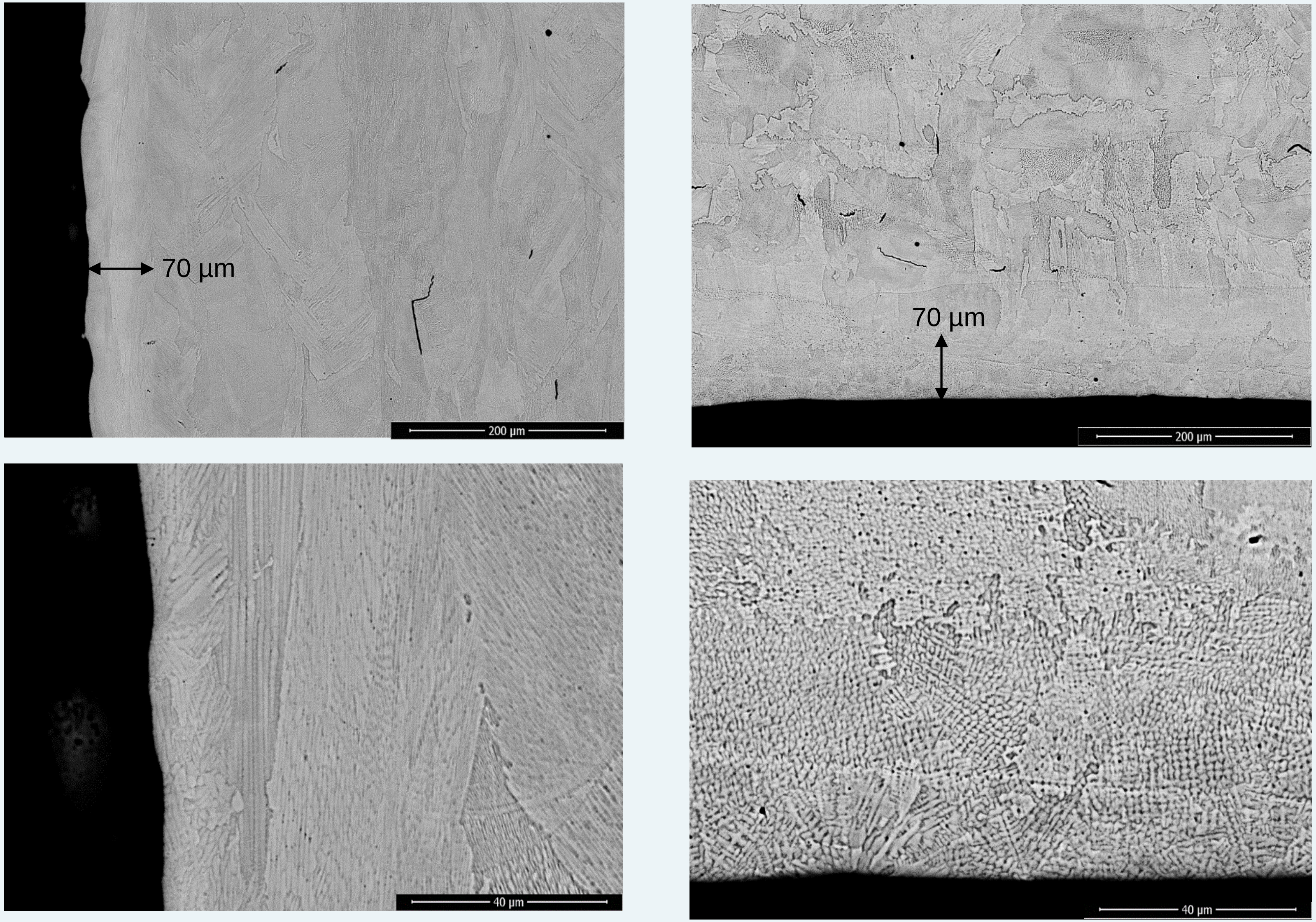


As-built surface :
+10% increase in developed area compared to polished surface
Ra in the building direction: 2.4 µm

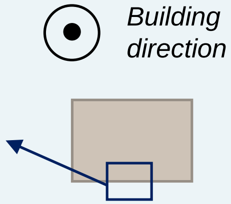
1. Material study – As-built characterization

Surface Microstructure – Cross-section





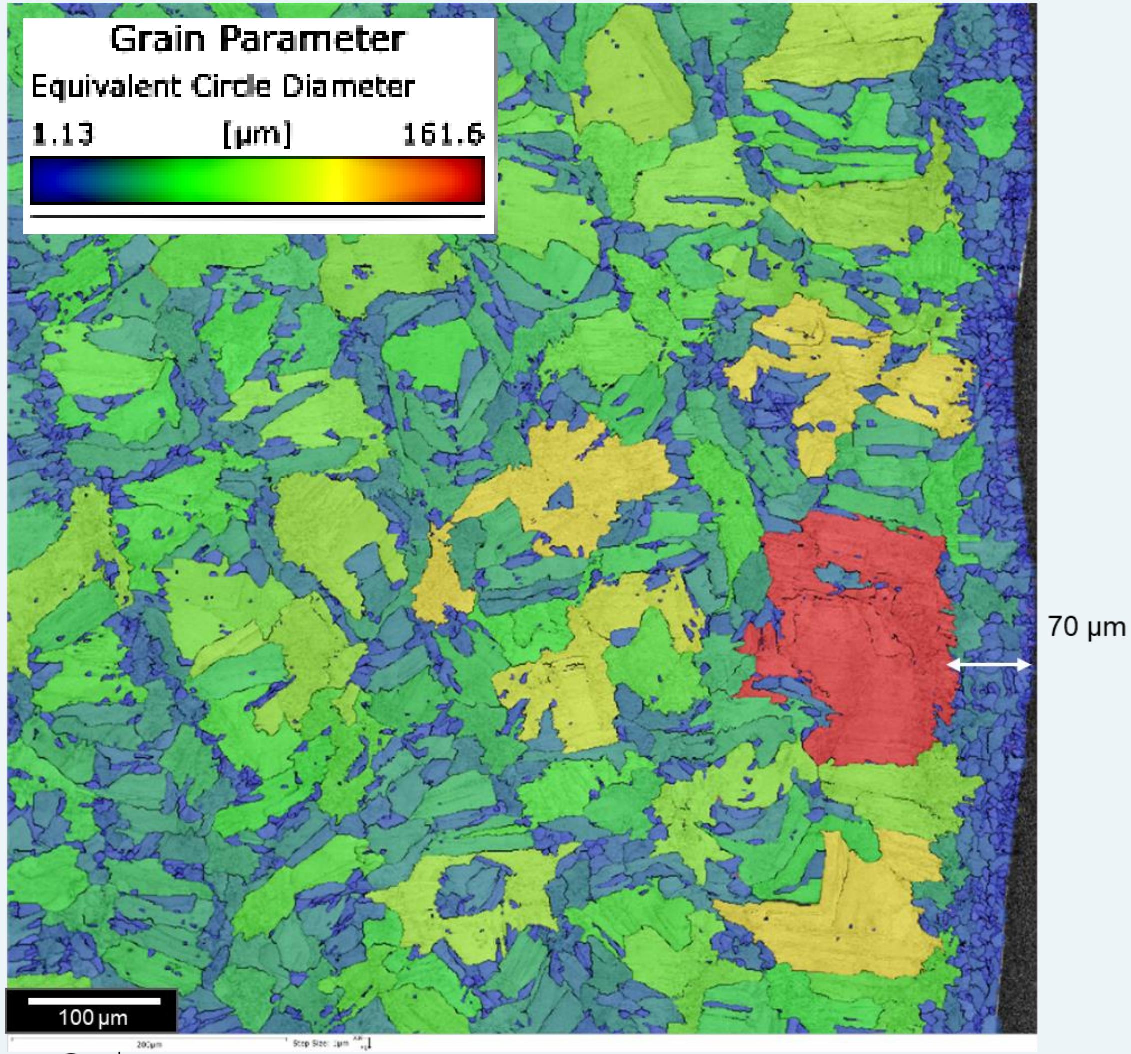
70µm shell microstructure differing from the columnar grains core microstructure → due to processing



Dendrites size ≈ 1.1 µm

1. Material study – As-built characterization

Surface Microstructure – Cross-section



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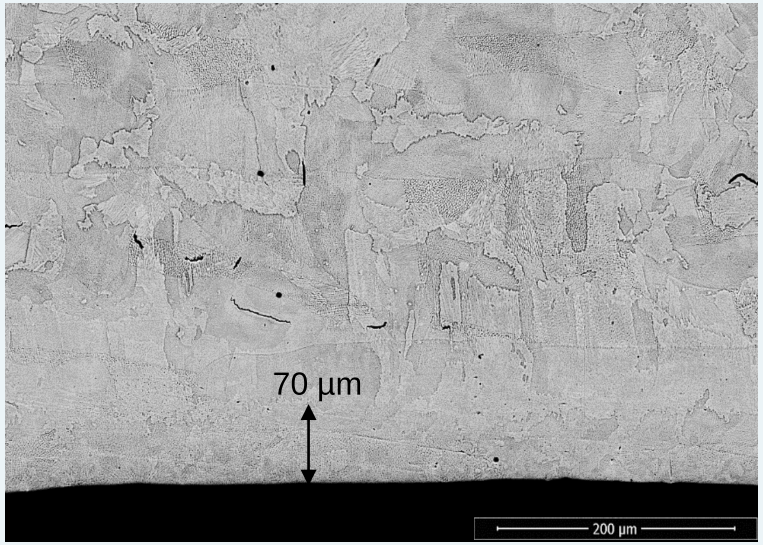
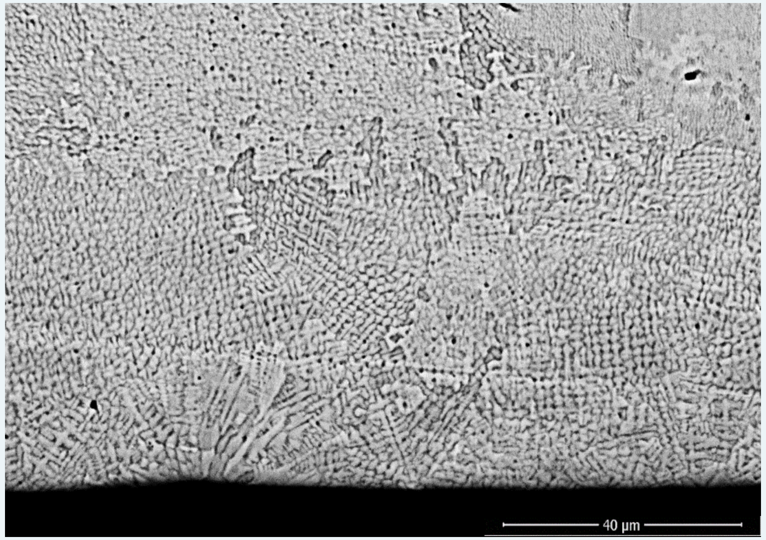
EBSD mapping





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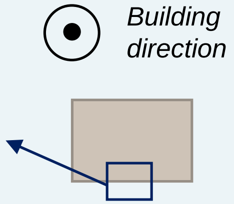


Dendrites size ≈ 1.1 µm

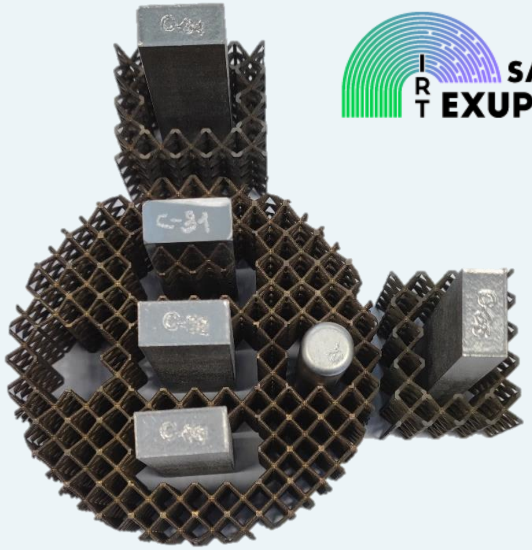
2. Experimental strategy

2. Experimental strategy

HIP conditions:

- Dwell at a supersolvus T° / High pressure / static Ar 4.8
- **Unknown gas impurity composition**
- **Closed system**

As-built surface *Polished surface*

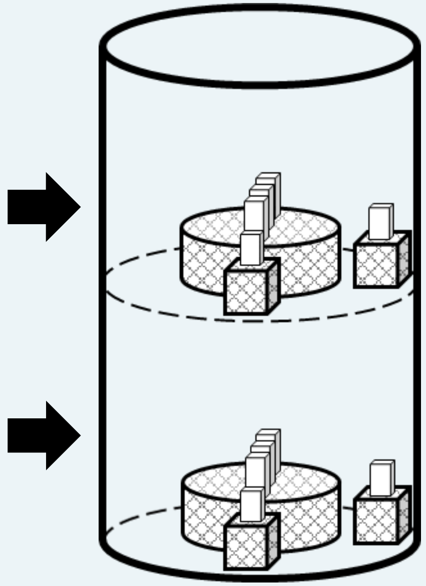


Specimens + Preoxidized 718 alloy holders put together

Parametric study:

- **Position in the chamber**
- **Surface condition: fully polished or As-Built surfaces**
- Different HIP chambers

→ outcome most likely dependent on the charge



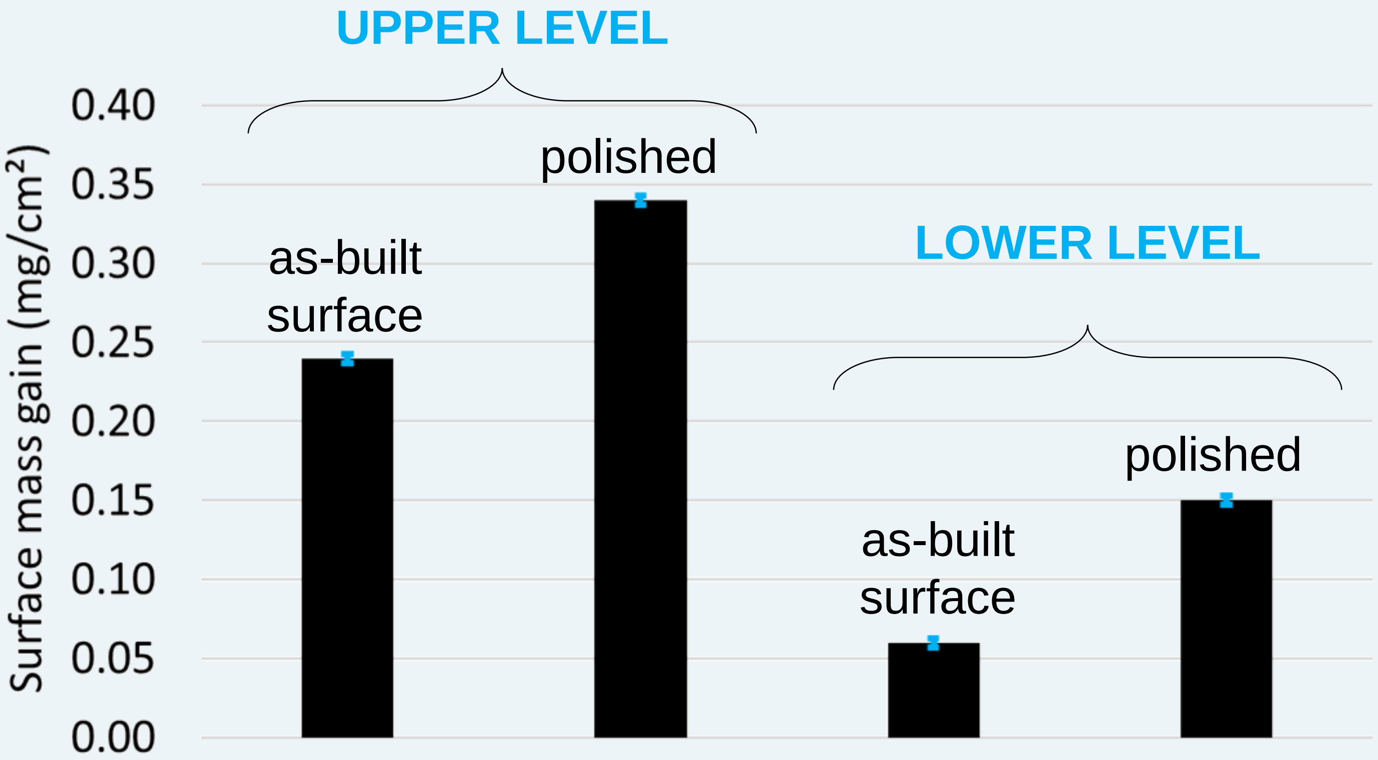
Sample positionning in the HIP chamber

3. Oxidation characterization

3. Oxide characterization: Mass gain data after HIP

Ante-HIP

Post-HIP

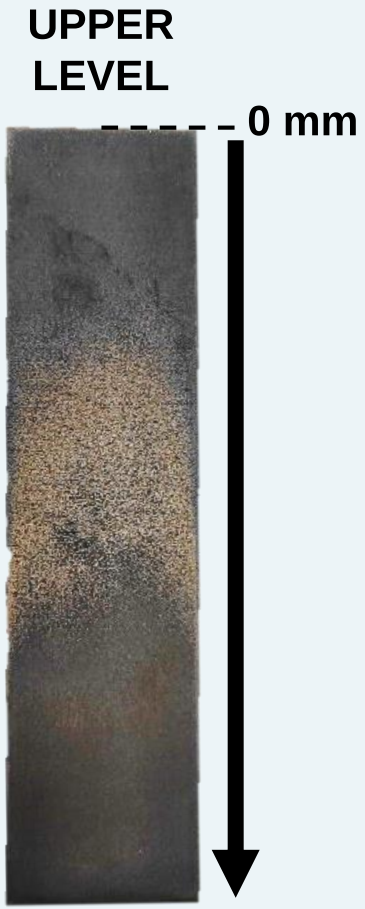


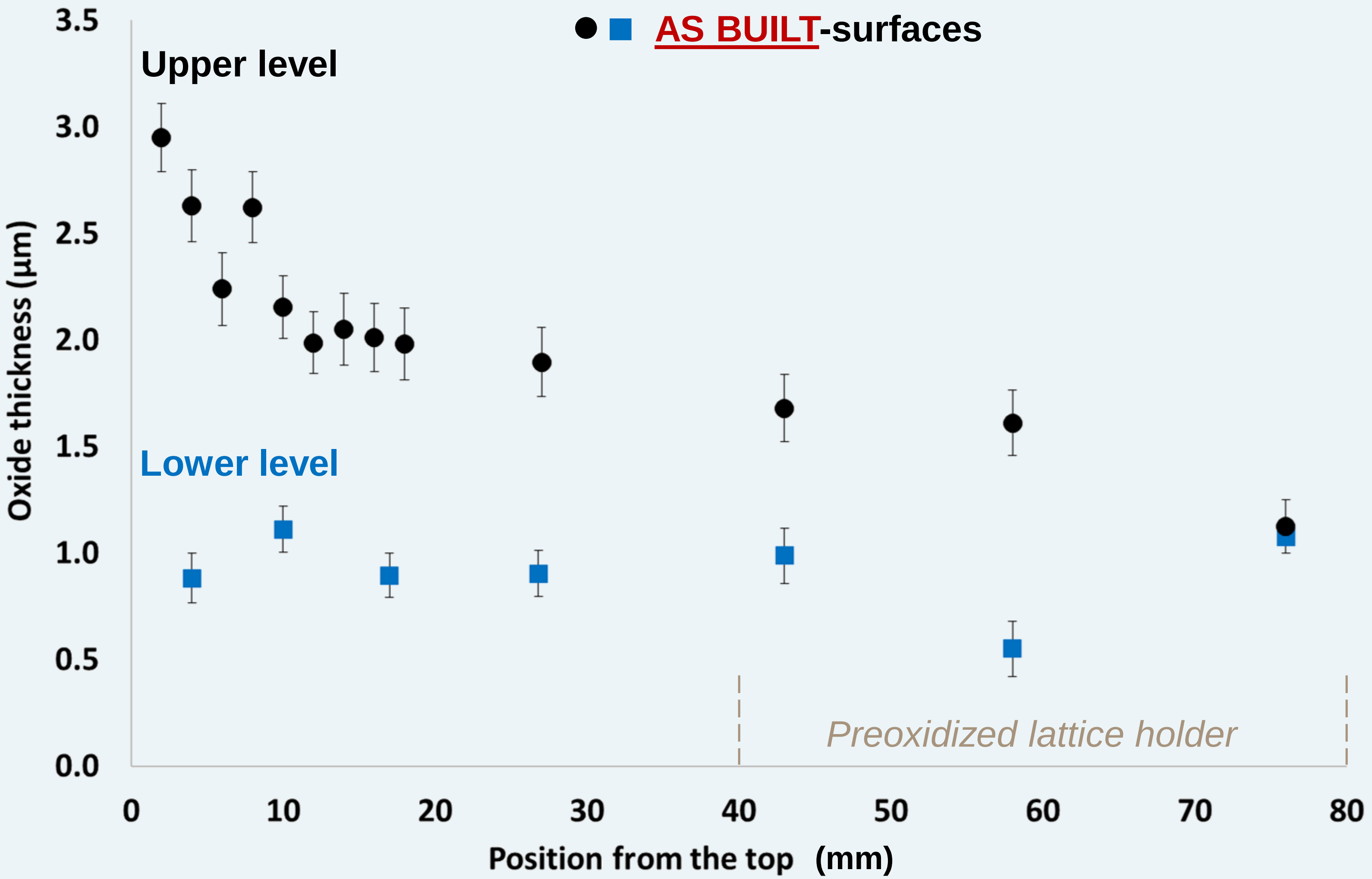
Surface mass gain of specimens after HIP in Ar

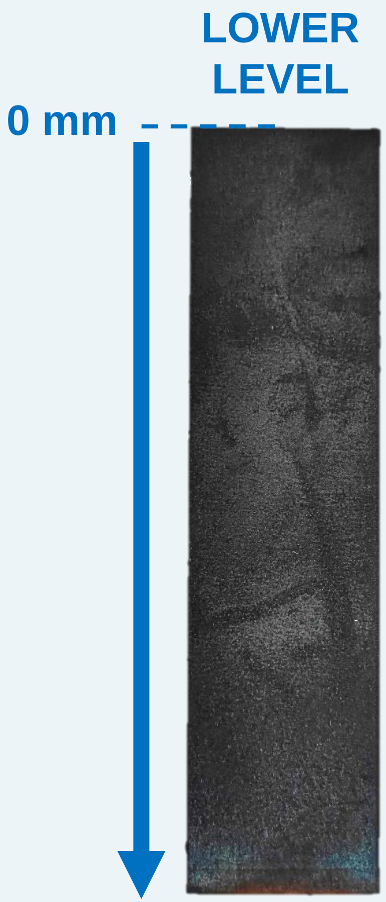
Upper HIP level + Polished surfaces → higher mass gain

Lower mass gain on as-built surfaces: most likely spalling

3. Oxide characterization: Cross-section analysis – Oxide thickness



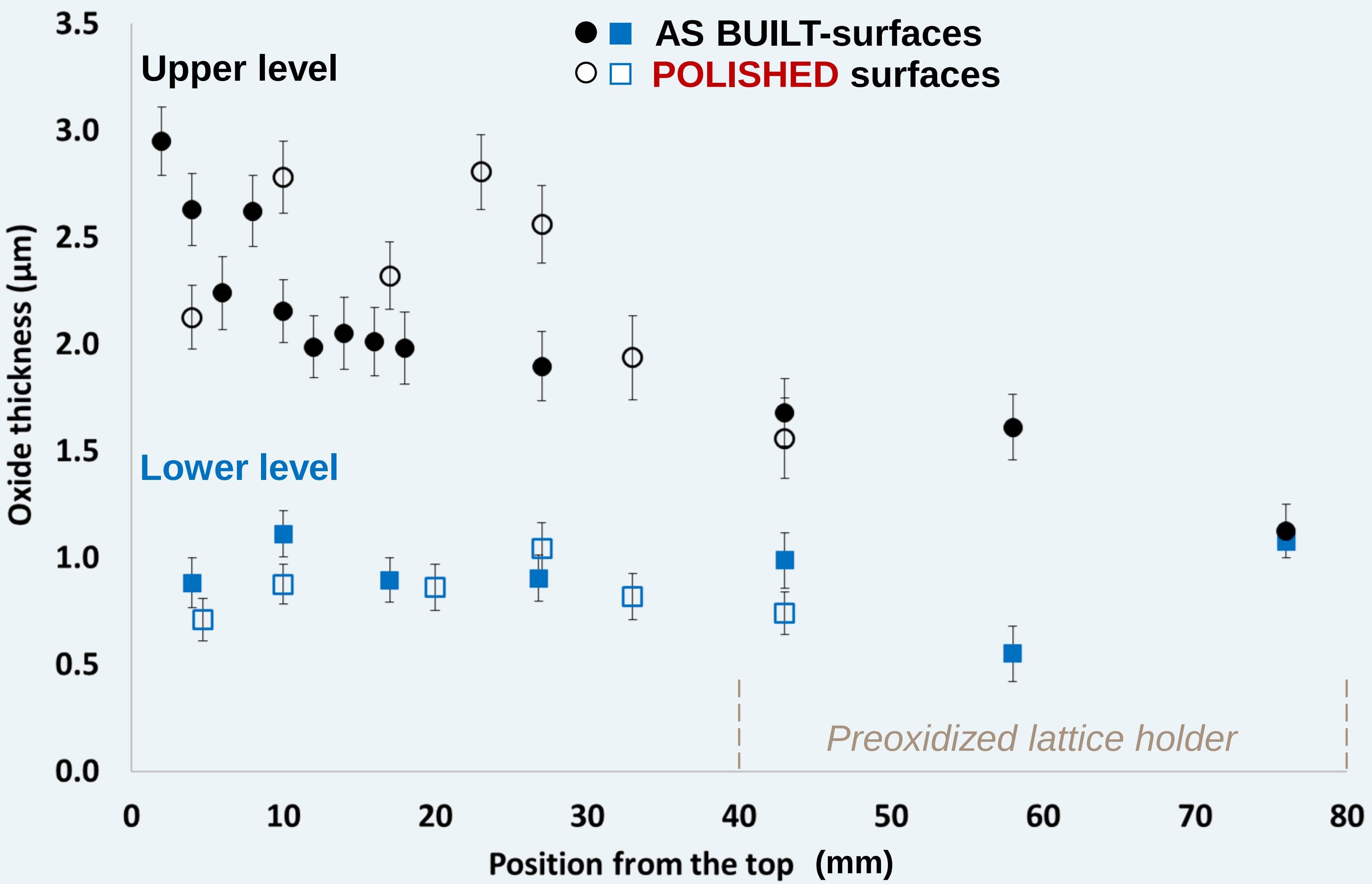




3. Oxide characterization: Cross-section analysis – Oxide thickness

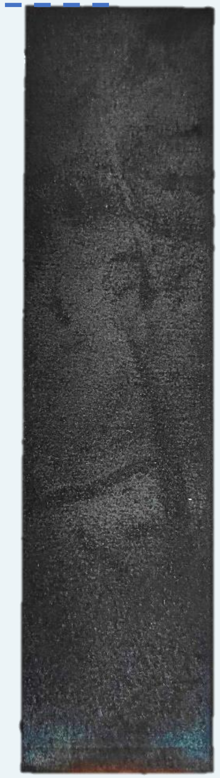
UPPER LEVEL

0 mm



LOWER LEVEL

0 mm



- **No thickness gradient** on the **LOWER** LEVEL surface specimens
- **Thickness gradient** on the **UPPER** LEVEL surface specimens + thickness in the **upper** level **>** thickness in the **lower** level:

Gradient of partial pressure of oxydant in the chamber ?

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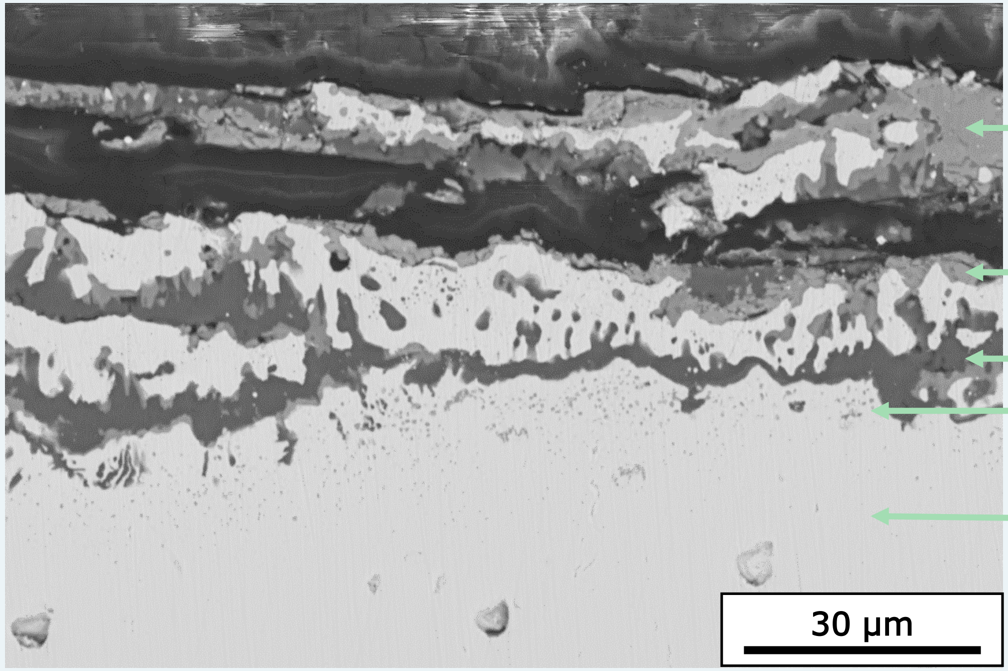
3. Oxide characterization: Oxide nature

Oxide identification:
EDS + XRD analysis (+XPS)

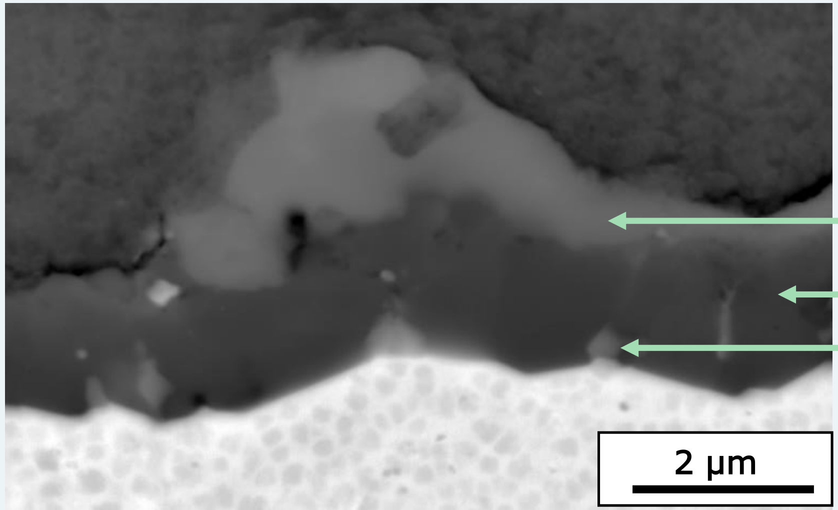
HIP parameters / 1 bars of laboratory air



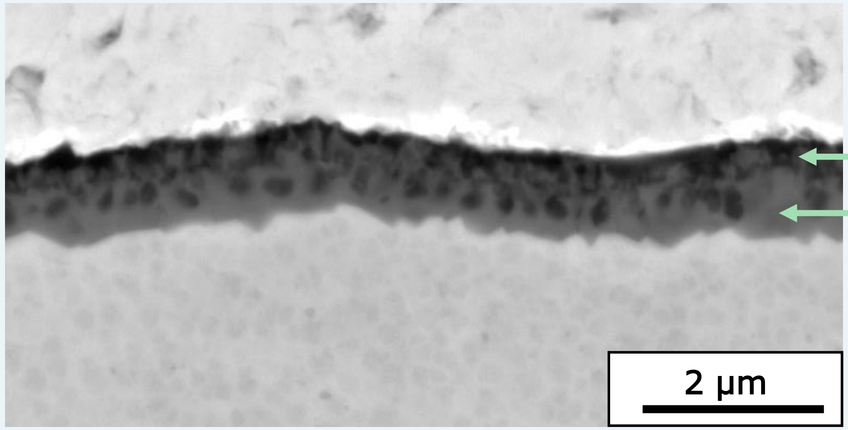
HIP conditions / 1000 bars of Ar

HIP UPPER LEVEL (as-built)

SEM-BSE



HIP LOWER LEVEL (as-built)



High temperature oxidation of the Ni-based superalloy:
- **Chromia-forming in air**
- **but Alumina-forming in HIP + Ti-rich oxides/nitrides/carbides layers and precipitates**

Conclusions

Conclusions

AM γ-γ' nickel-based superalloy specimens are HIPed in inert Argon 4.8
However, **some oxidation, nitridation and even carburation is identified**.

- An oxide nature and thickness **gradient along the position in the chamber**
  → There could be a **gradient of oxidant partial pressures in the HIP chamber**

- **HIP conditions** enable **the formation of nitrides, oxinitrides or carbonitrides layer**
  + the change from chromia-forming behavior in air to alumina-forming in HIP

ON THE GASEOUS ENVIRONMENT

- The Ti nitrides should grow for **$P_{O2} < 10^{-19}$ atm** according to thermodynamical calculations

→ *Hypothesis on HIP environment:*

local oxygen gradient at the specimen surface

and/or

strong influence of the H content on the P_{O2} value

MERCI !

Questions ?

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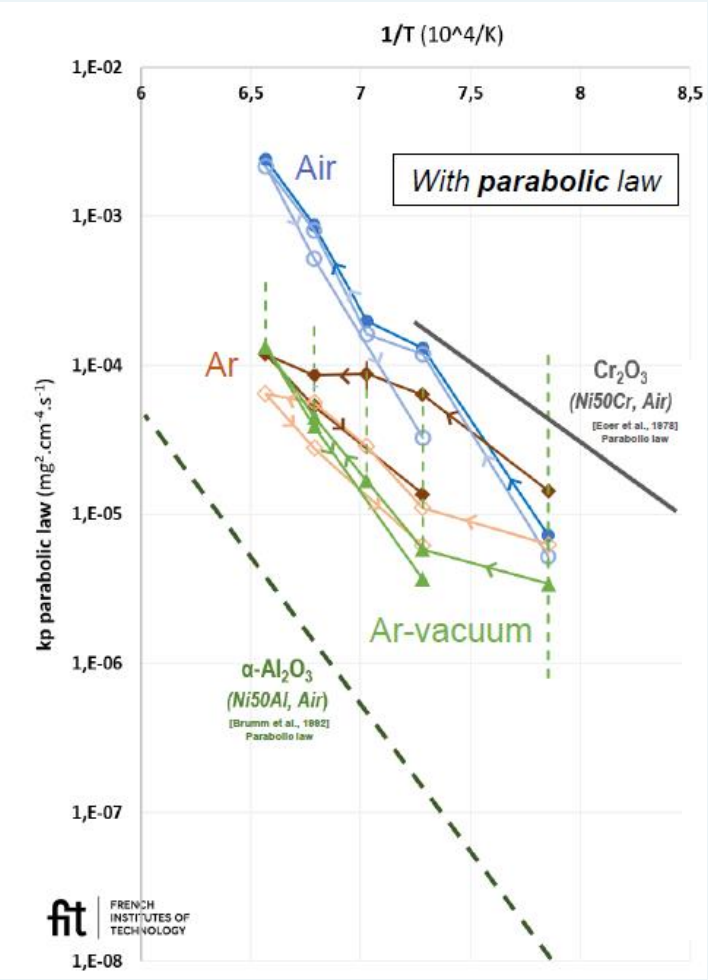
Outlooks

- Stepwise multi temperature Thermogravimetric Analysis
  - To assess oxidation/nitridation kinetics
  - Different environments used: Air, Ar, vaccuum+Ar leak



Arrhenius diagram after SMT-TGA between 1000°C and 1250°C

- Reproduction of the oxide/nitride layers obtained with HIP but in a conventional furnace at 1 bar
  - To be able to readily predict the oxide/nitride growth in HIP