Compaction isostatique à chaud d'alliages à base de Nickel

Focus sur l'oxydation et la nitruration

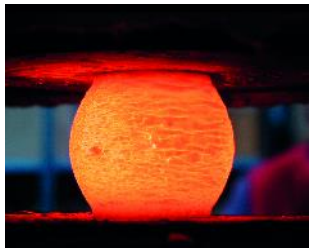
Aurélien PRILLIEUX

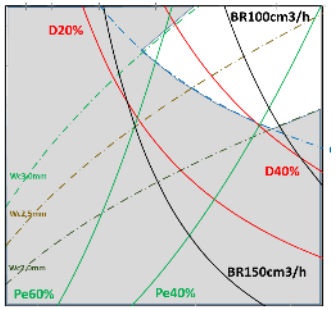
Journées Annuelles du GDR COnCOrD

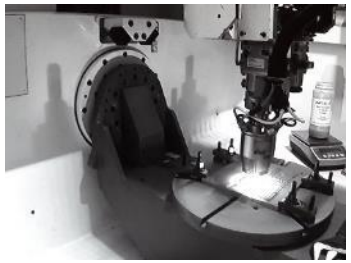
09/10/2025

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IRT Saint Exupéry - MMP:



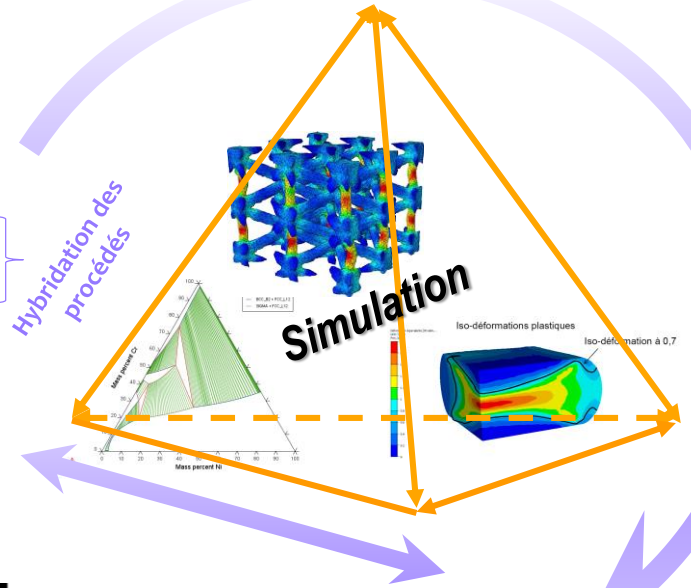




Procédés

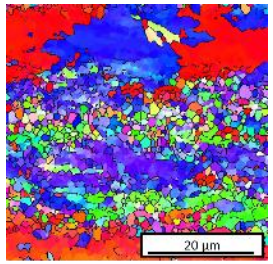
Fabrication additive
Forgeage net-shape
FA pour réparation
Contrôle en fabrication
Traitement thermique

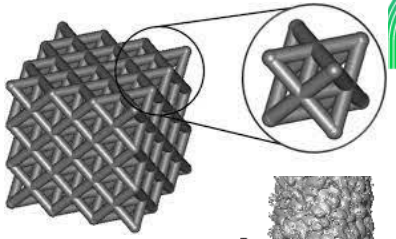
Hybridation des procédés

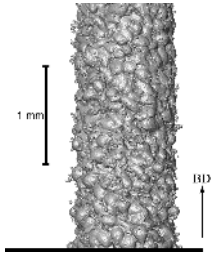


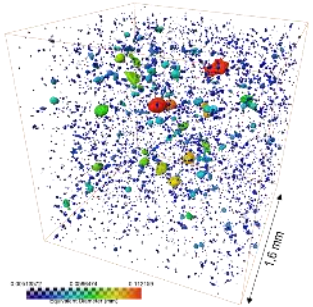
Matériaux

Caractérisations multi-échelles
Transformations de phases
Etude des défauts de FA



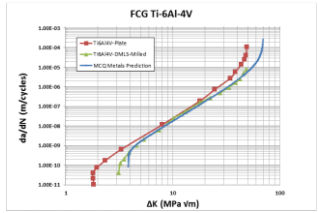


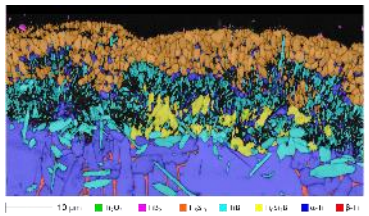




Propriétés

Résistance à la fatigue et aux dommages
Effet des défauts sur les propriétés mécaniques



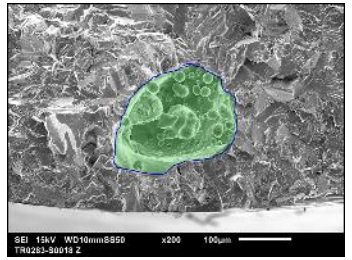




Performance

ACV
Cout





FRENCH INSTITUTES OF TECHNOLOGY

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PF technologiques de l'IRT:

2 moyens DED-LB modulo 400 BEAM

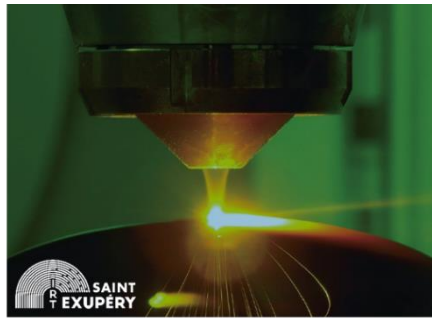
Fil





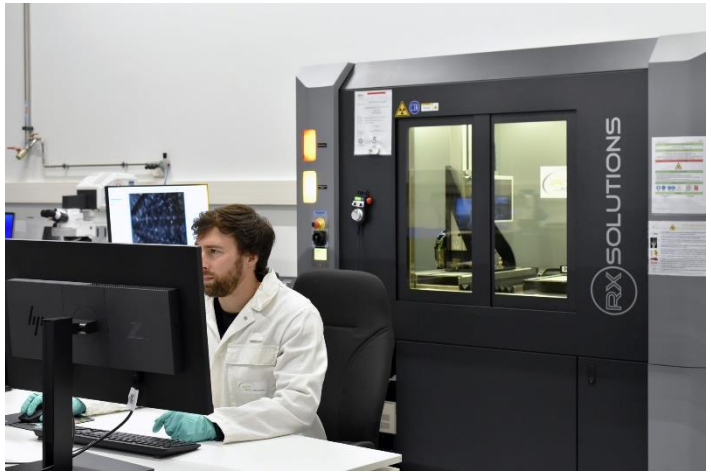
Poudre







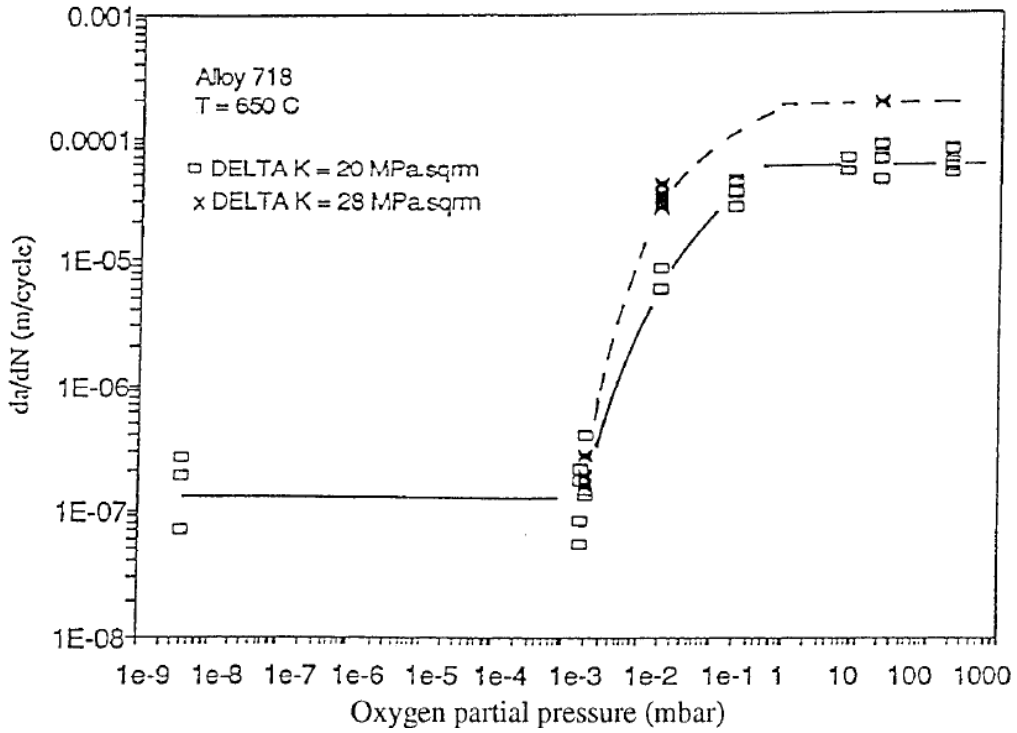
CIC Quintus



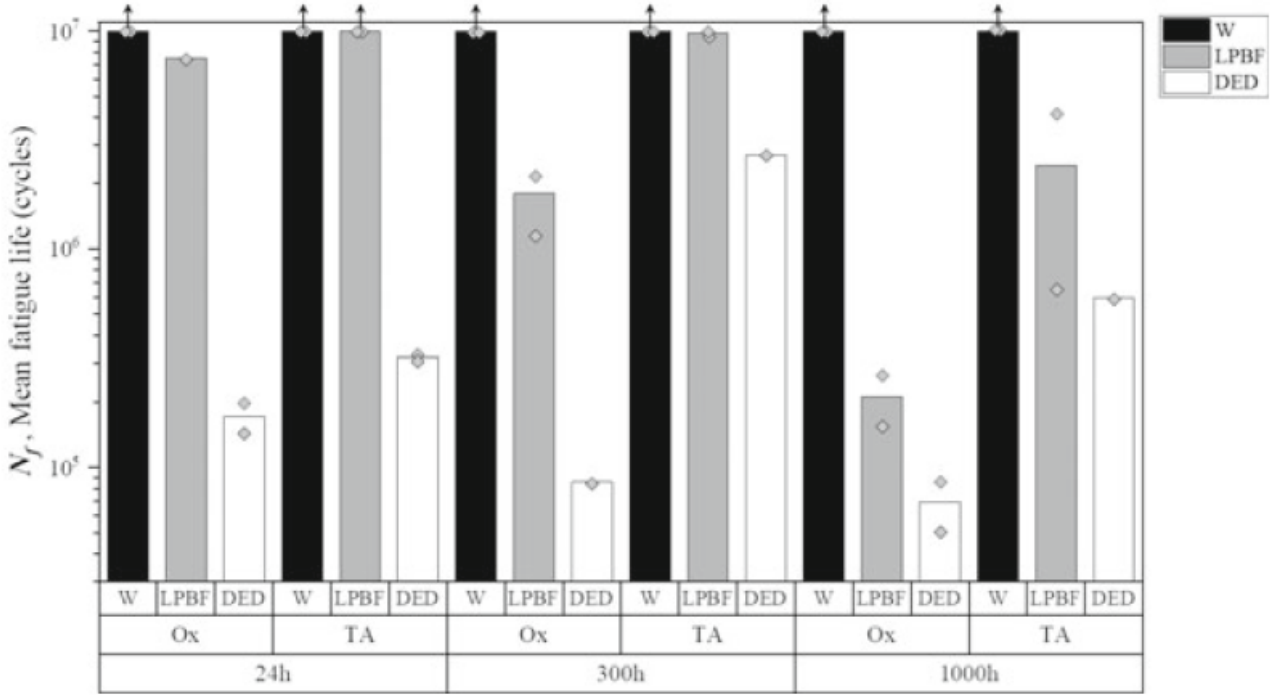
Tomographe – X Rx solution

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Etude du couplage mécanique oxydation



Effet de la P_{O2} sur propagation de fissure, 718 *[1]*



Comparaison de la vie en fatigue après oxydation de matériaux issus de différents procédés, 625 *[2]*

[1] ANDRIEU, *et al. Oxidation mechanisms in relation to high temperature fatigue crack growth properties of alloy 718*. *Superalloys*, 1994, vol. 718, no 625, p. 619-631.

[2] DE LEON NOPE, Grace, *et al.* Effects of High-Temperature Oxidation on Fatigue Life of Additive-Manufactured Alloy 625. In : *TMS Annual Meeting & Exhibition*. Cham : Springer Nature Switzerland, 2023. p. 249-269. de FA, 625

Phénomènes d’oxydation lors de cycles CIC

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Enjeux industriels :

Altération de certaines propriétés des matériaux après un cycle CIC

- **Perte** de capacité d'assemblage de pièce : étapes de soudage ou brasage post-CIC
- **Diminution** de certaines propriétés mécaniques, **fatigue,** avec l'ajout d'un CIC dans les cycles de TTh

Projet HIPPOME (2021-2023)

Air Liquide | CIRIMAT Toulouse | SAINT EXUPÉRY | SAFRAN SAMC

Etude de la **modification** de **l'état de surface** des pièces lors d'un **cycle CIC**

MROZOWSKI, Nicolas, PRILLIEUX, Aurélien, EPIFANO, Enrica, *et al.* Surface reactivity during a hot isostatic pressing treatment of Ni-based superalloy specimens manufactured by laser powder bed fusion and metal inject molding. *Corrosion Science*, 2024, vol. 234, p. 112119.

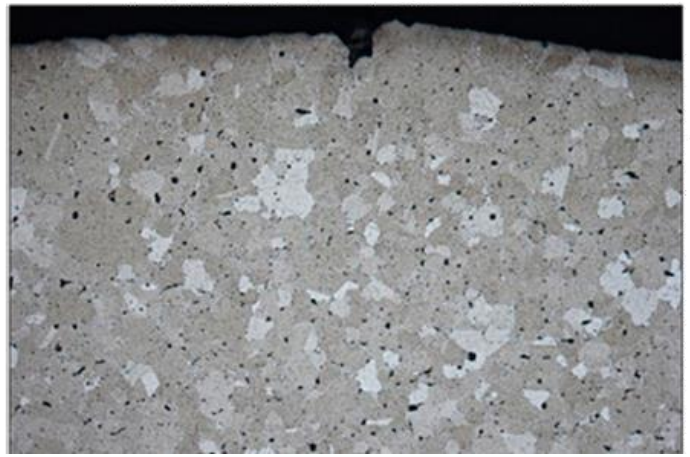
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Rôle du CIC :

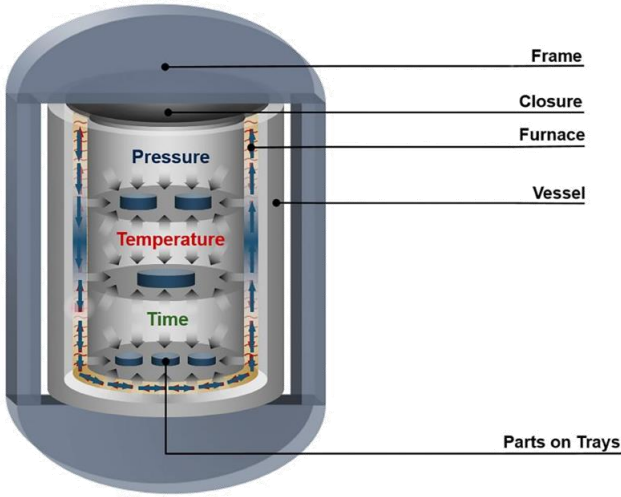
- Métallurgie des poudres, FA
  → défauts : fissures, pores

- CIC → fermer les pores, consolidation du matériau :
  - Très haute température (>1000°C)
  - Haute pression totale (≥100 MPa)
  - Gaz inerte Argon

- Cependant, traces impuretés gazeuses (O_2, N_2, H_2O…) détectées dans les fours CIC



Métallurgie des poudres CM247LC avec haute densité de pores

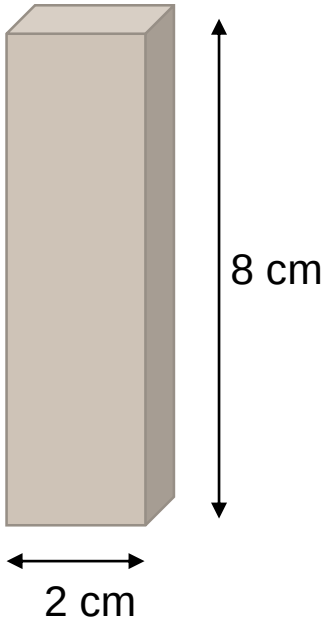


Etendue du phénomène d'oxydation lors du CIC?
Identification de la source des impuretés

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Alliage base-Ni γ/γ'

Barreaux d'alliage base-Ni γ/γ' fabriqués par **LPBF**



Durcissement par **γ' – $Ni_3(Al,Ti)$**



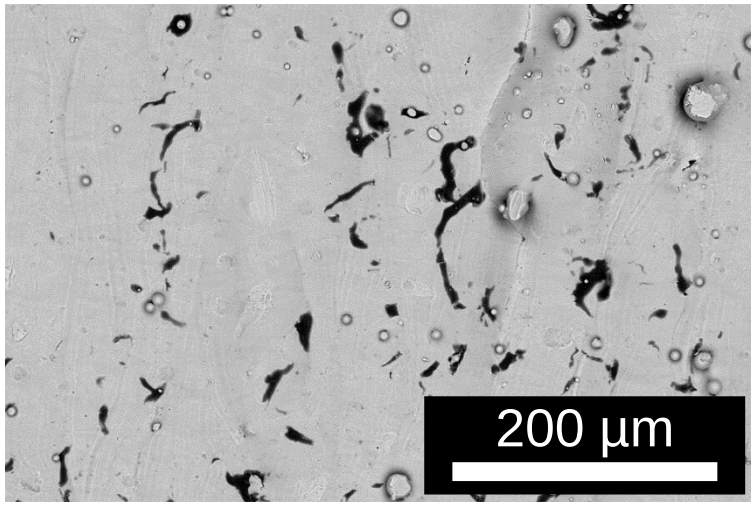
CIC IRT Saint Exupéry

Conditions d'essai :1200°C / 4h / 1000 bars / Ar4.8

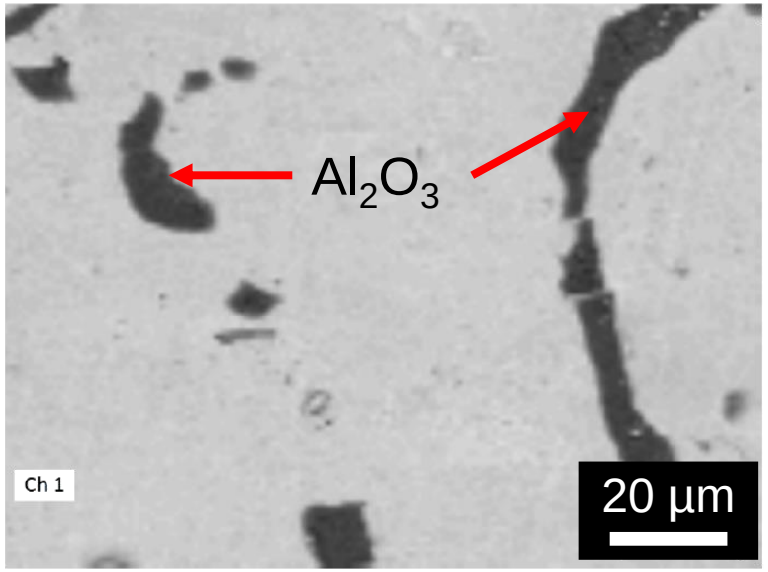
N.B. Composition de gaz inconnue

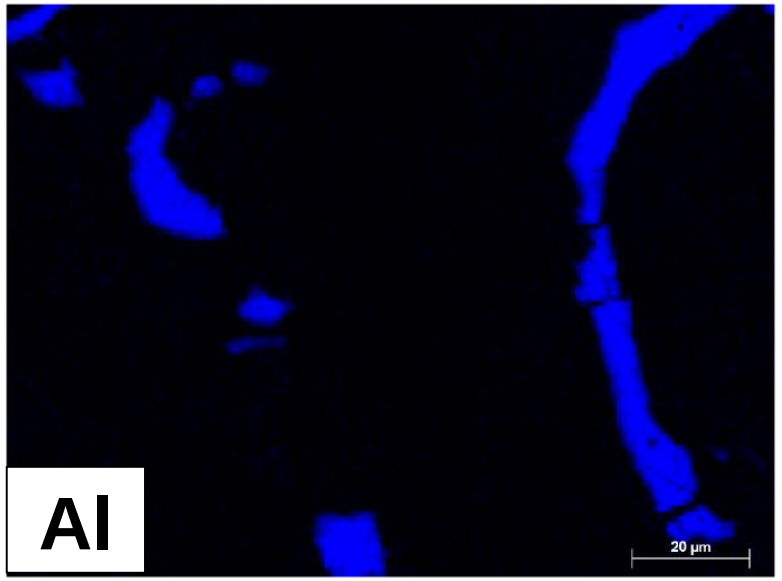
Surface « brute de fab »

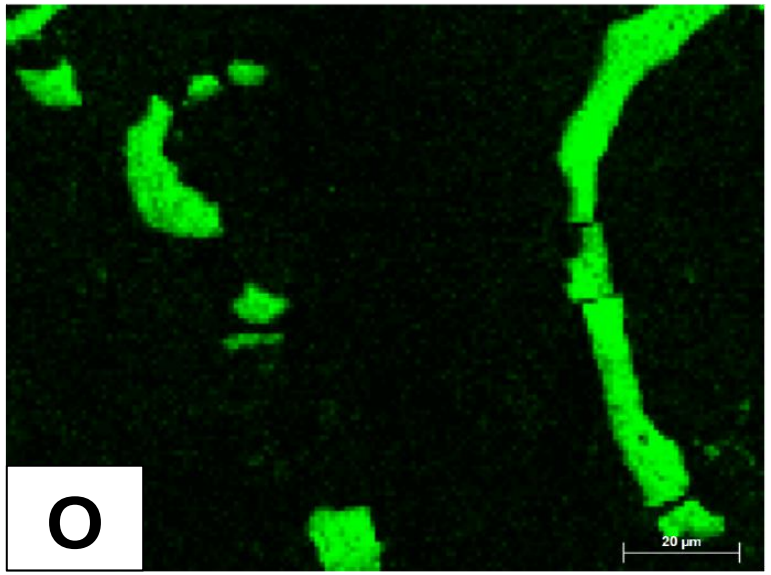
SEM BSE



Direction de fab



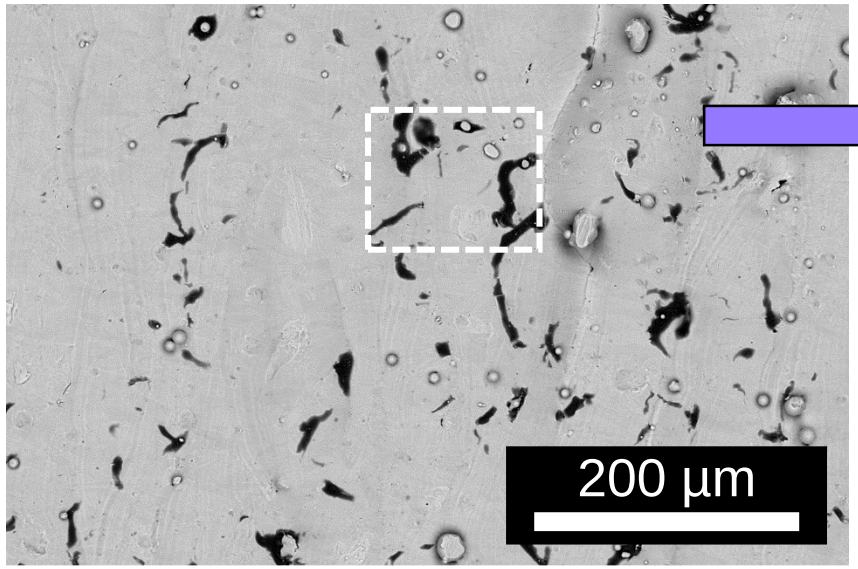




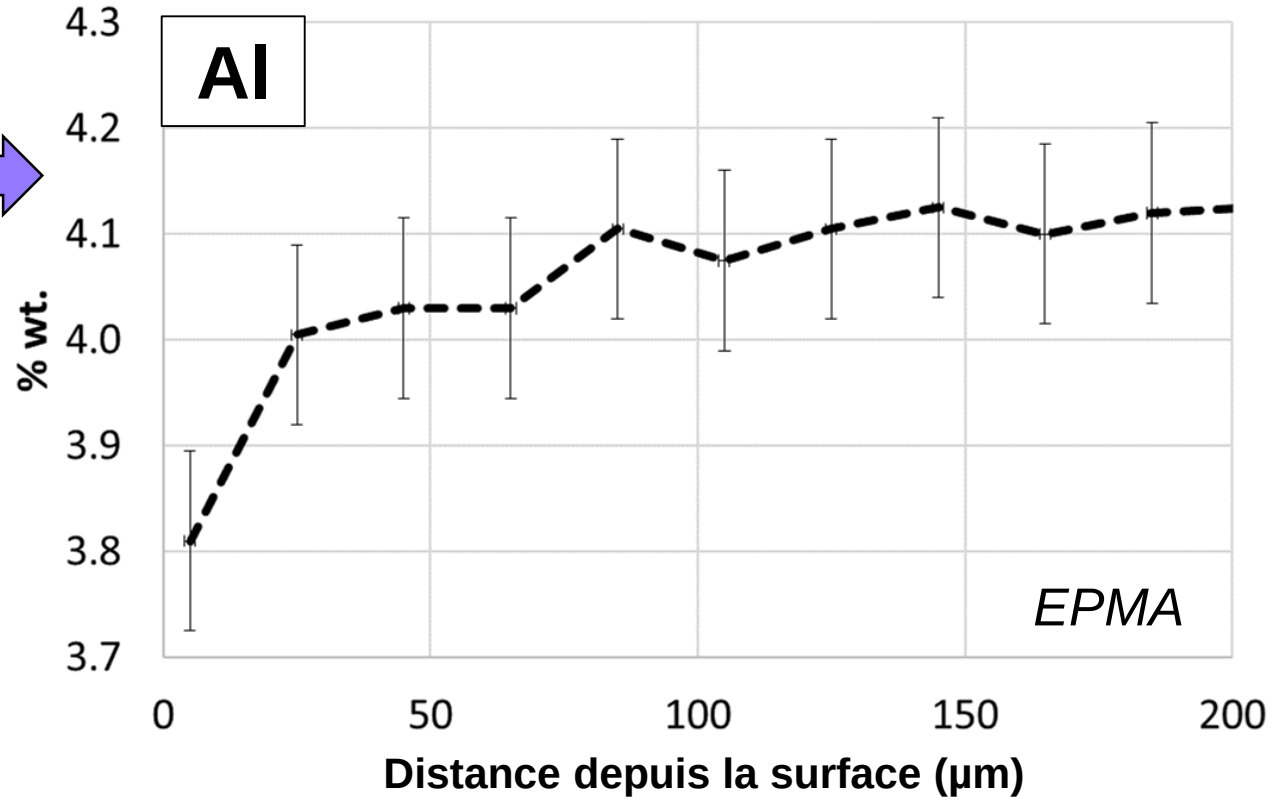
Oxydation lors du procédé LPBF

Surface « brute de fab »

SEM BSE



Direction de fab



Formation d'Al_2O_3

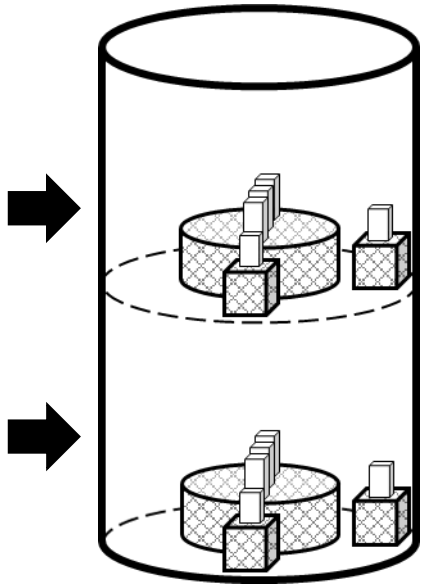
↳ Légère déplétion en Al proche surface

Effet sur phase durcissante γ' - $Ni_3(Al,Ti)$

Effet du traitement CIC

Etude paramétriques :

- Position dans le four
- Etat de surface – polie ou « brute de fabrication »



Position des échantillons dans le four CIC



Brute de fab *Polie*

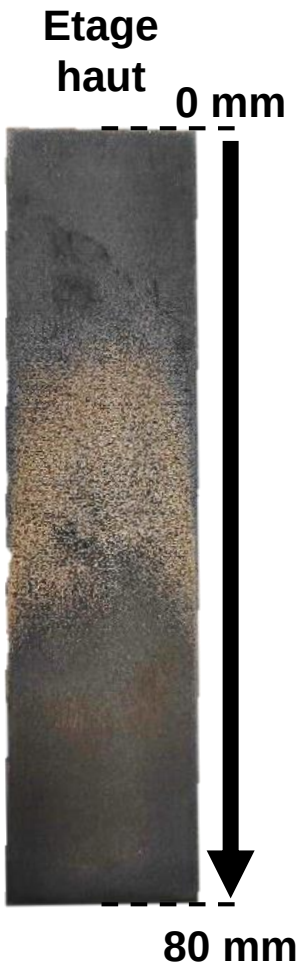
Caractérisation des oxydes :

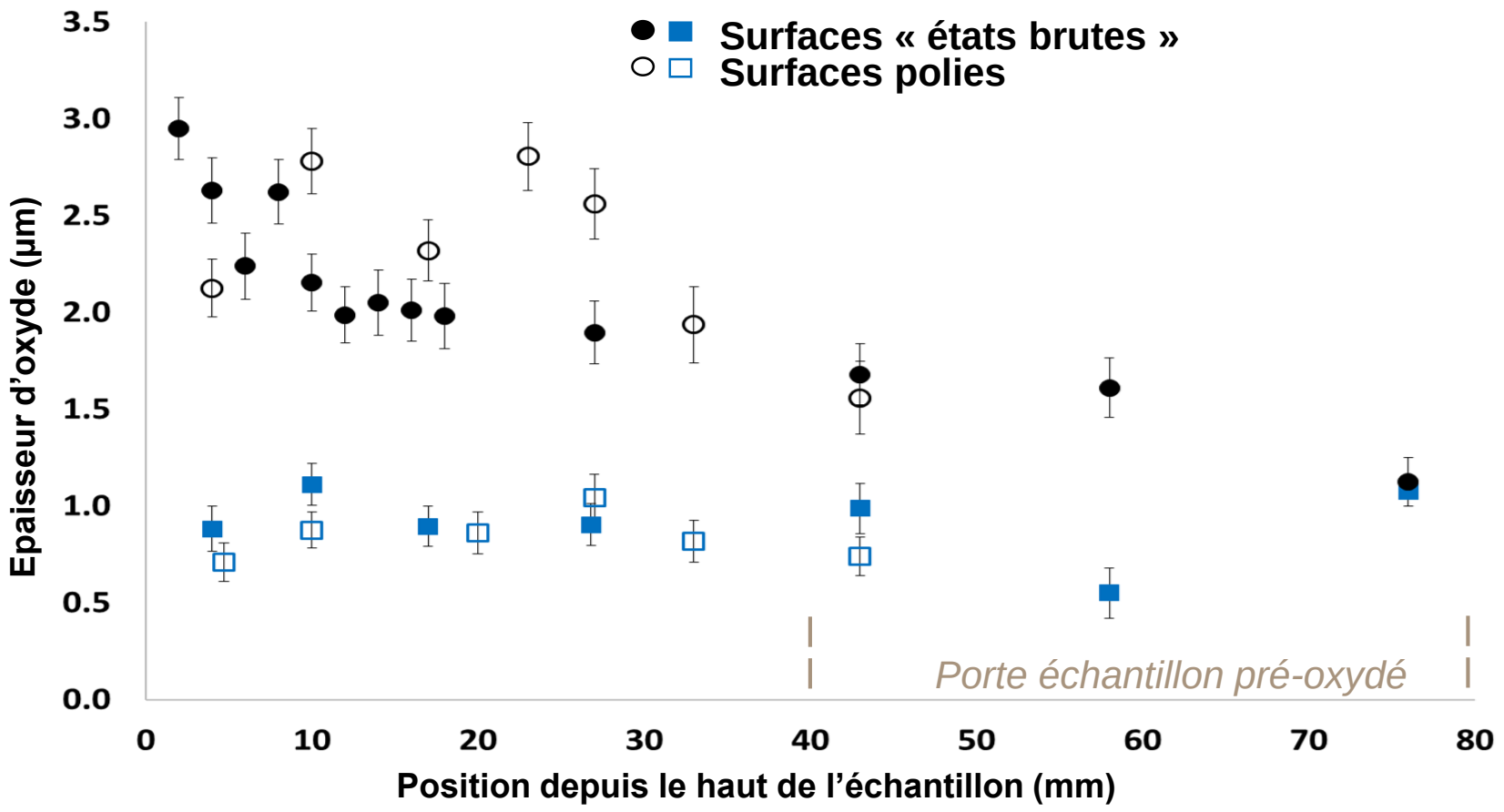
- Gain de masse
- Coupe transverse MEB + EDS
- MET



Echantillons +
Porte échantillon en 718 pre-oxydé

Effet du traitement CIC



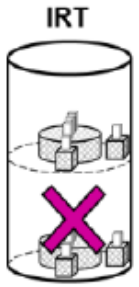


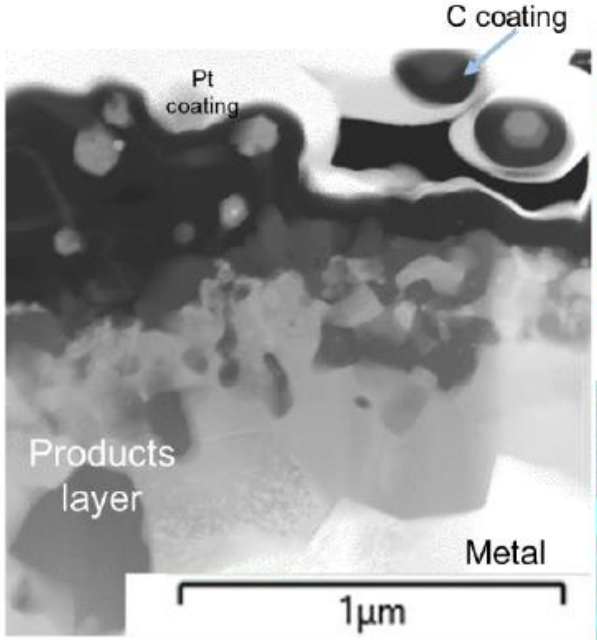


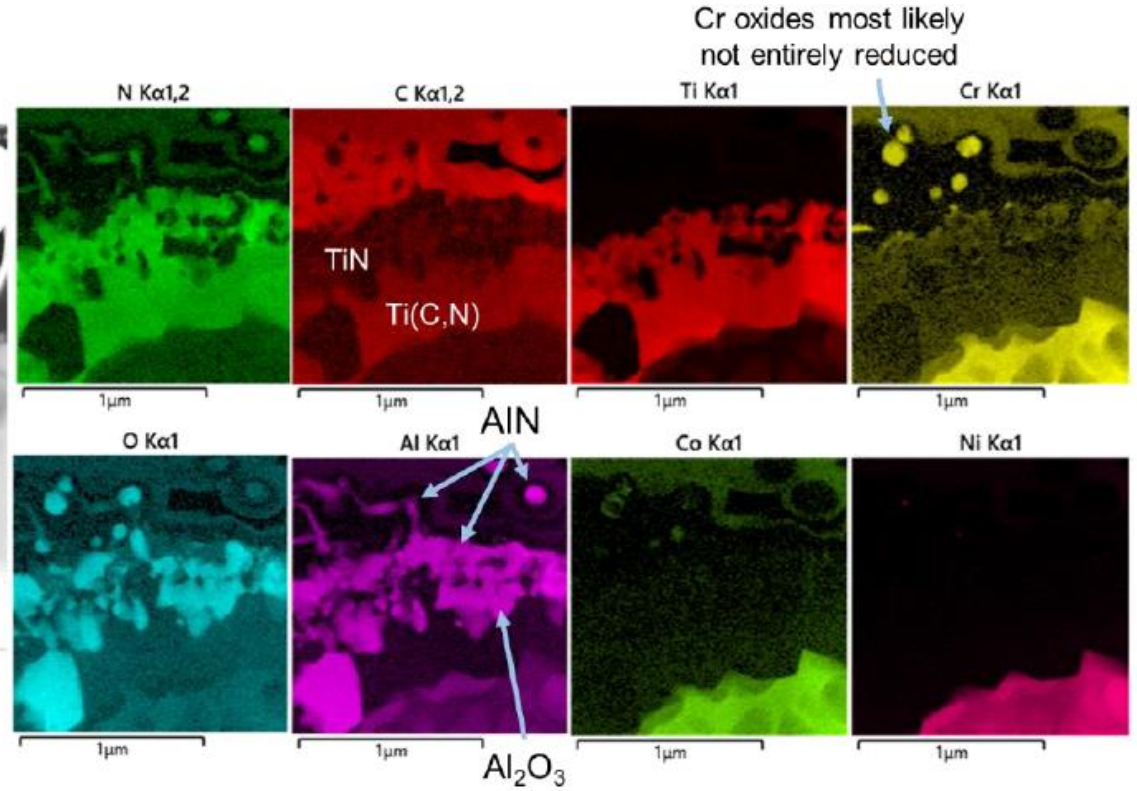
- **Pas de gradient d'épaisseur** sur les échantillons **à l'étage bas**
- **Gradient d'épaisseur** sur les échantillons **étage haut**
- Epaisseur étage haut **>** Epaisseur étage bas

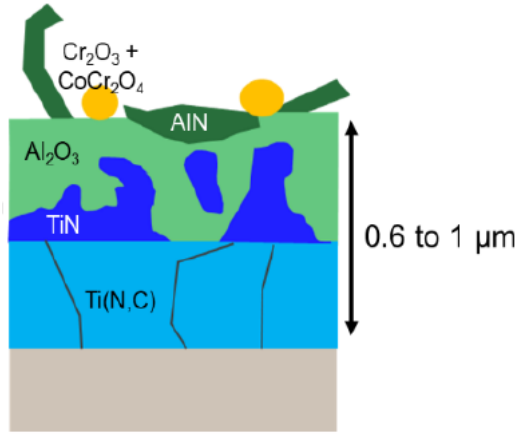
Gradient $P_{(O,N)2}$ gradient dans le four ?

Effet du traitement CIC



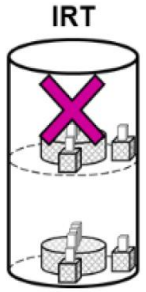


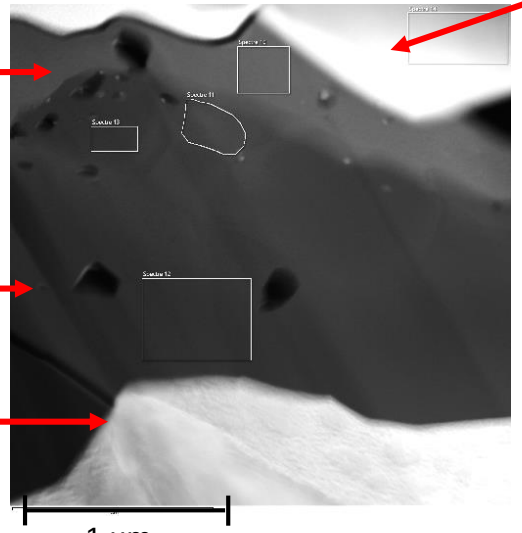


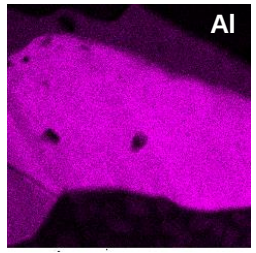


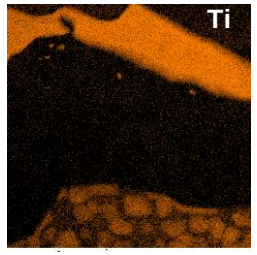
- Couche continue de **Ti(C,N)** à l'interface métal/TiX
- Depuis la couche de **Ti(C,N)** vers la surface :
  - **TiN** et **Al_2O_3**, aiguille d'**AlN** et petits précipités de **Cr_2O_3** et **$CoCr_2O_4$**

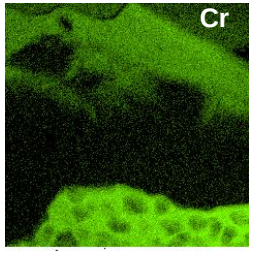
Effet du traitement CIC

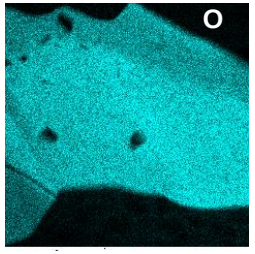


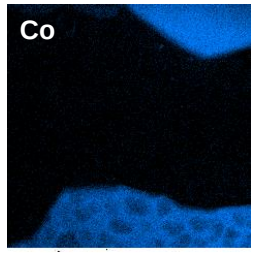


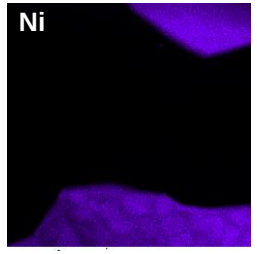


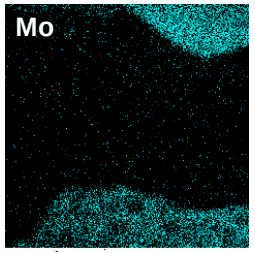












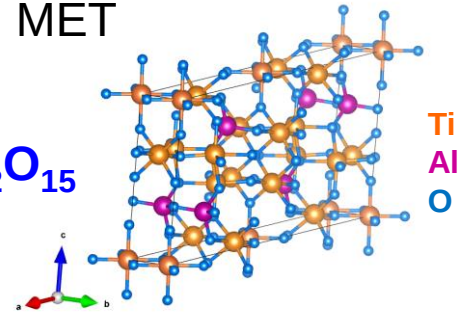
Composition oxyde-mixte :

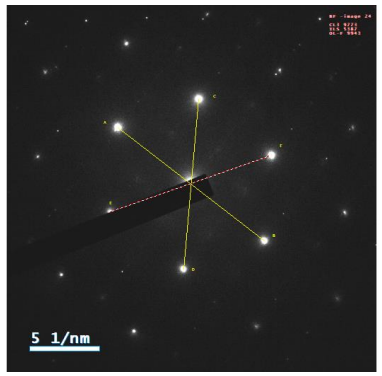
	Ti	Al	Cr	O
At.%	13.4	12.6	5.4	68.6

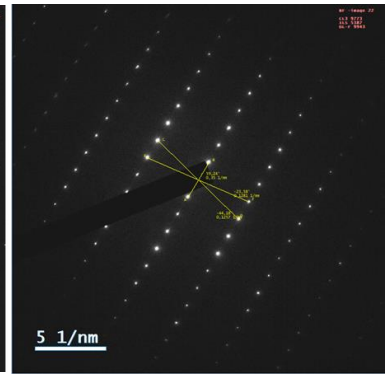
Plusieurs oxydes mixtes Ti-Al existent

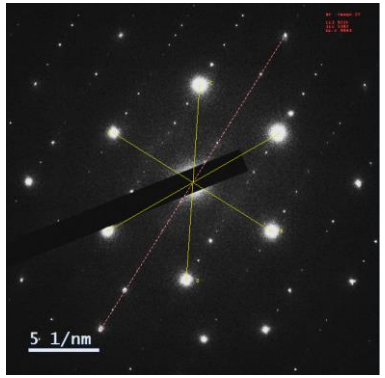
↳ Identification via analyses MET

↳ $Ti_7Al_2O_{15}$

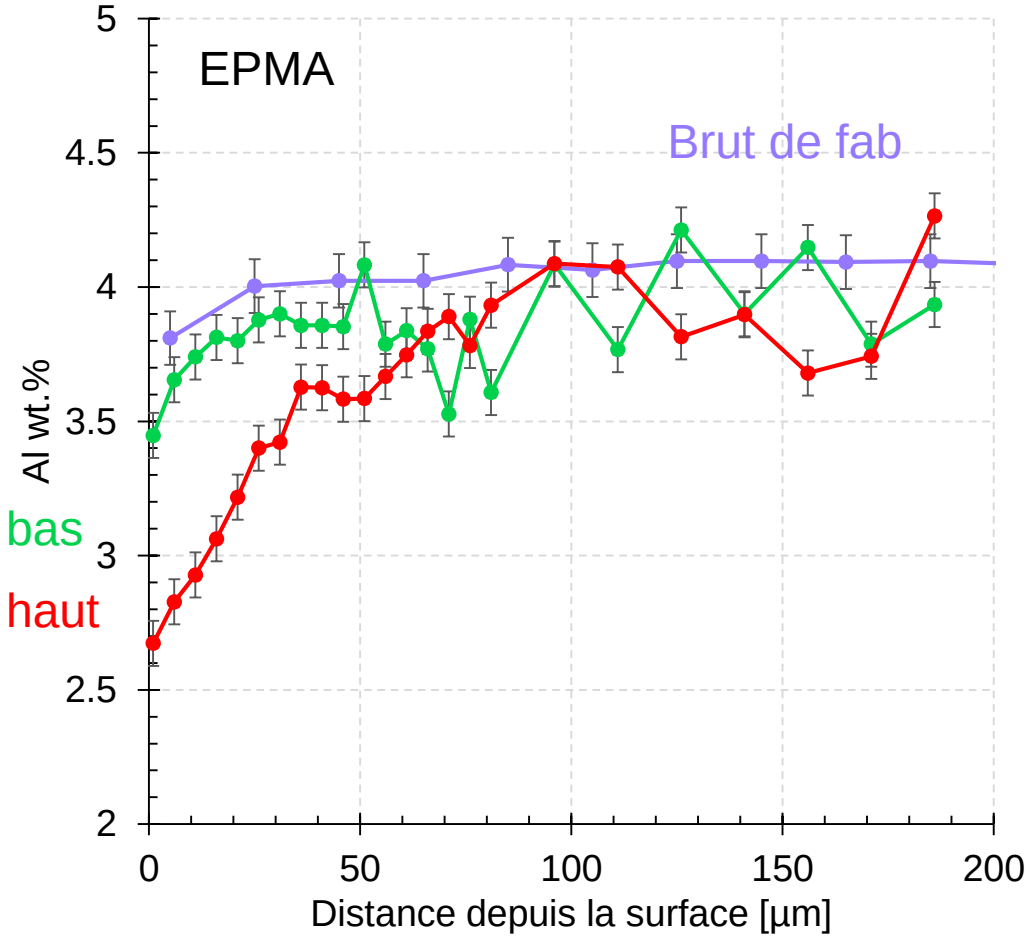


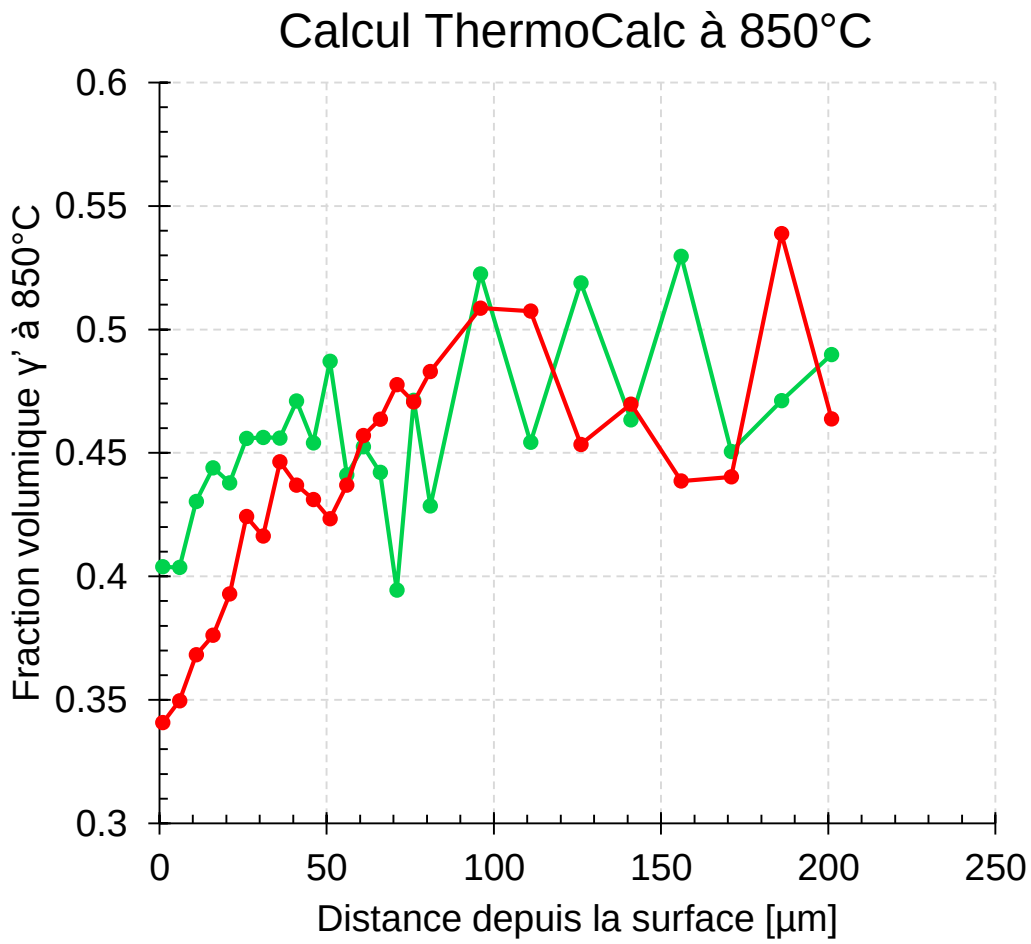






Effet du traitement CIC





Déplétion en γ' jusqu'à **100 µm** sous la surface pour les échantillons les plus oxydés

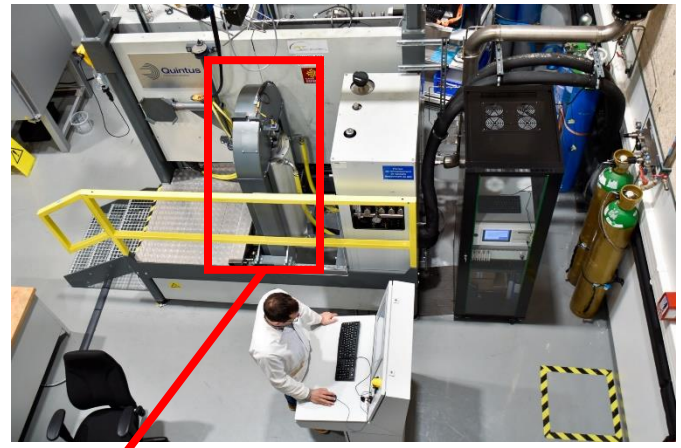
Moyen de contrôle de l’atmosphère

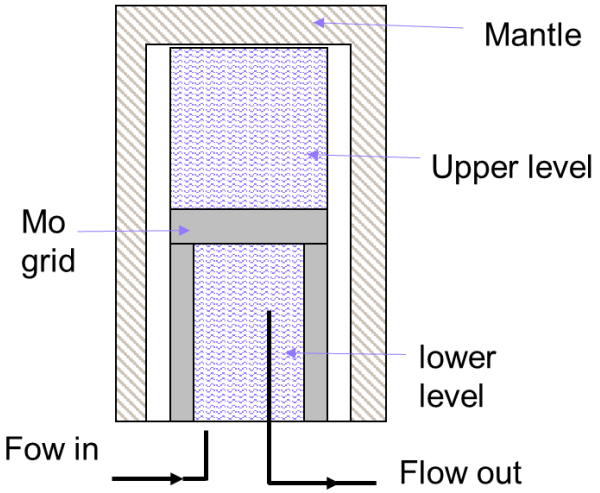
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Développement de moyen de contrôle d'atmosphère

Modification du CIC avec l'installation d'un analyseur de gaz

- **Quantifier** les impuretés
- **Défi technique** d'analyser un gaz à haute pression et haute température
- Point de prélèvement **au plus proche** des échantillons



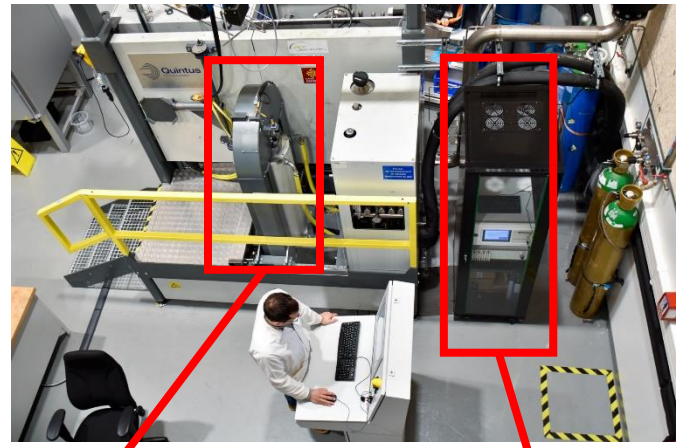


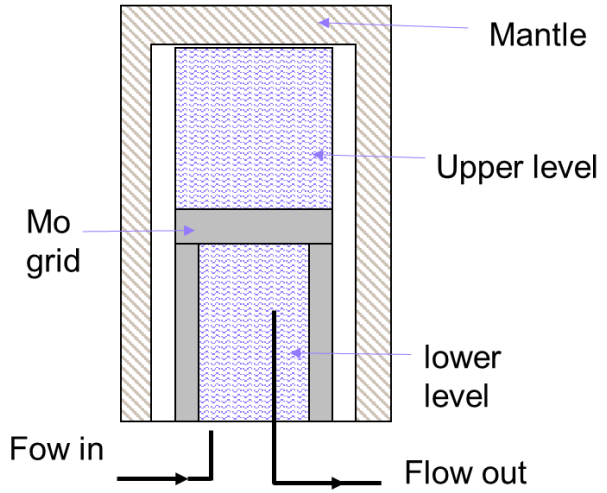
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Développement de moyen de contrôle d'atmosphère

Modification du CIC avec l'installation d'un analyseur de gaz

- **Quantifier** les impuretés
- **Défi technique** d'analyser un gaz à haute pression et haute température
- Point de prélèvement **au plus proche** des échantillons







Analyseur de gaz - Chromatographe Multidetek2

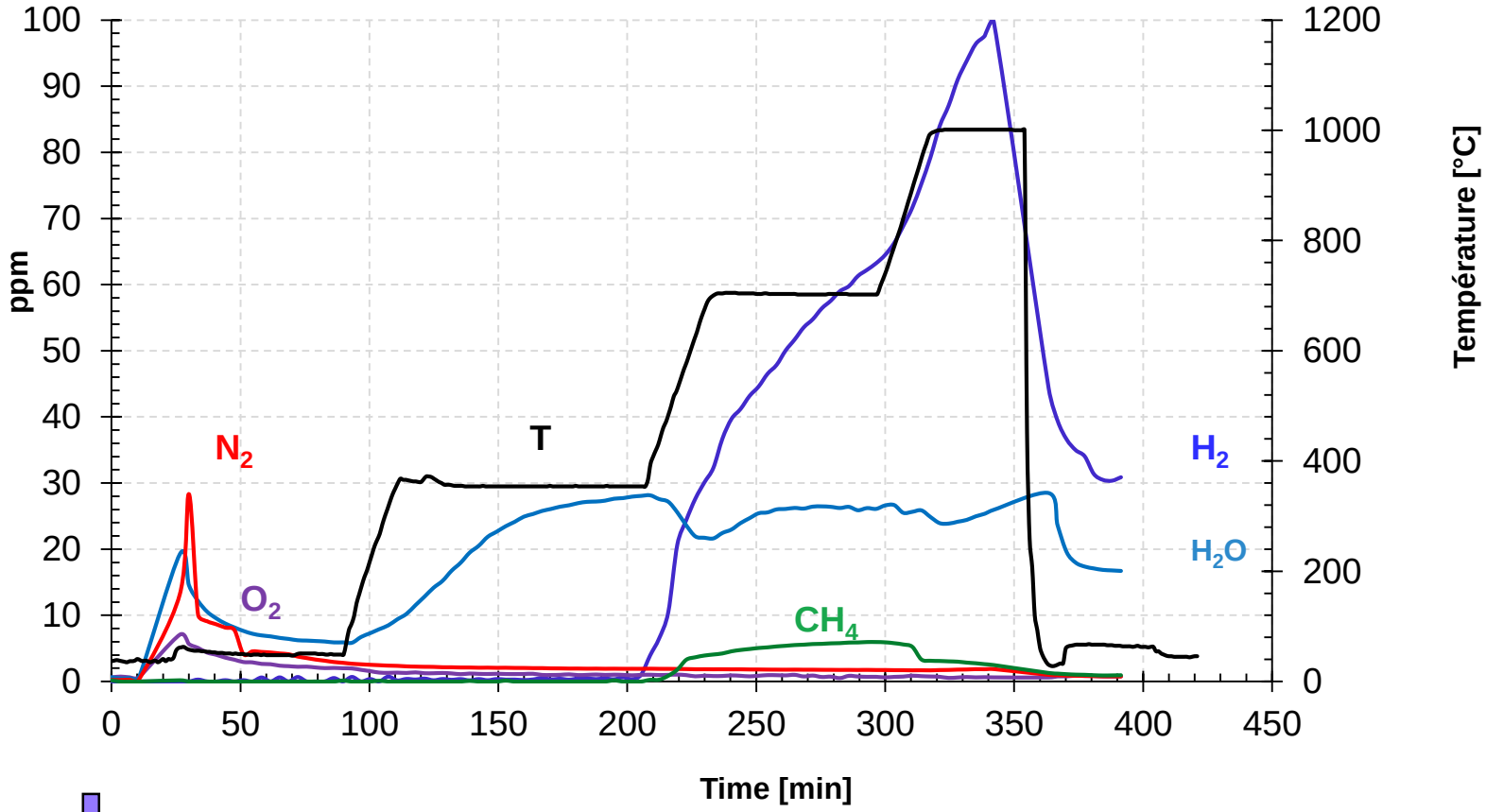
- N_2 → 100 ppb - 50 ppm
- H_2 → 100 ppb - 50 ppm
- CH_4 → 100 ppb - 50 ppm
- H_2O → microbalance à quartz 100 ppb-100 ppm
- O_2 → 100 ppb - 100 ppm

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Evolution de l'atmosphère au cours d'un cycle CIC

Evolution de la composition de l'argon en fonction de la température de palier

- Pas de charge
- Pression max 140 bar



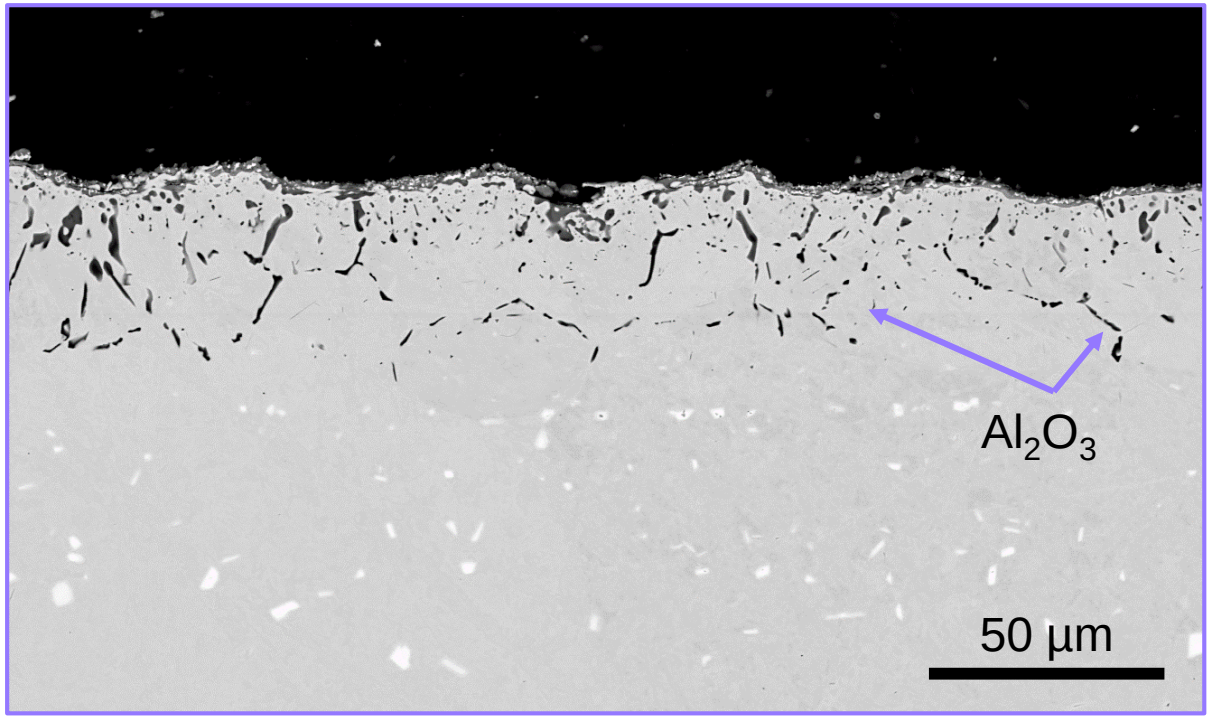
↳ Identification des espèces responsables de l'oxydation ➡ H_2O ➡ Solution getter

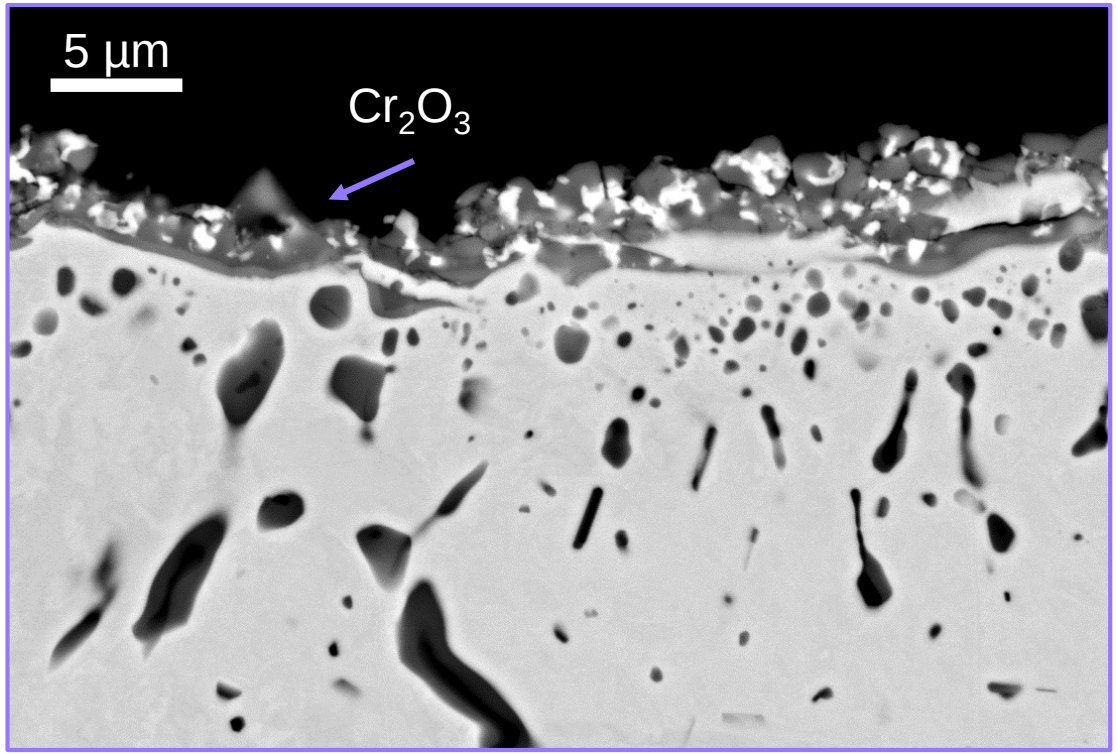
↳ Identification de températures critiques ➡ Procédure de nettoyage

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Développement d'un module getter

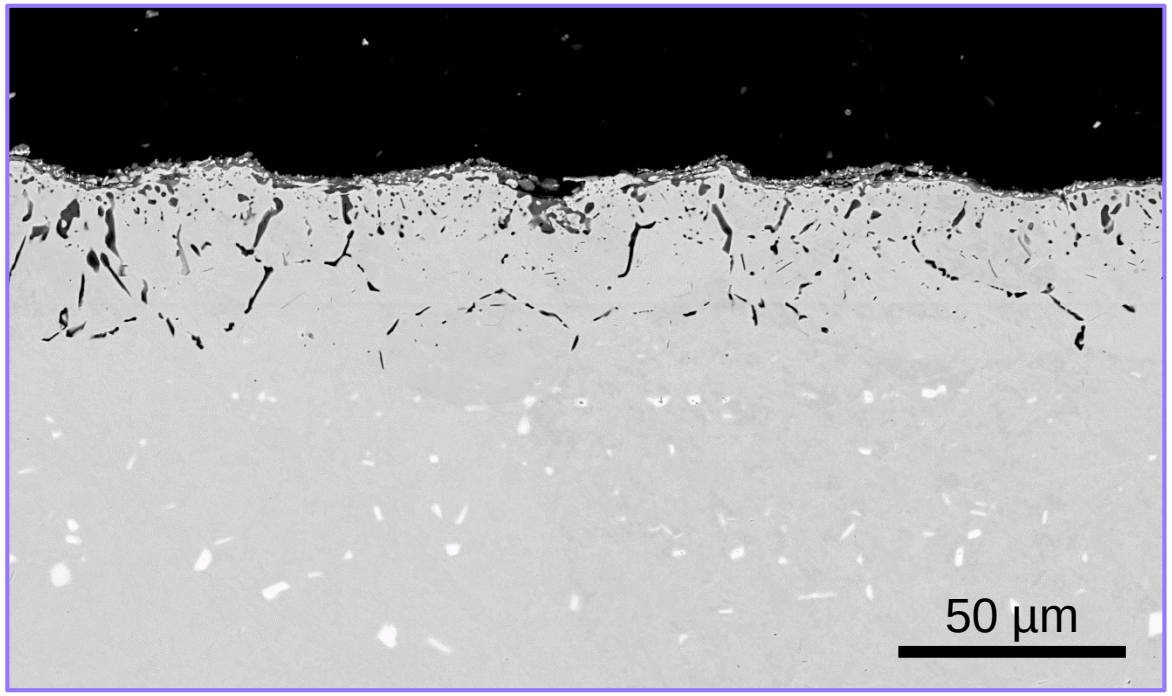
Observations MEB, 718 forgé
CIC 1200°C, 1000 bar, 4h, Ar 4.8

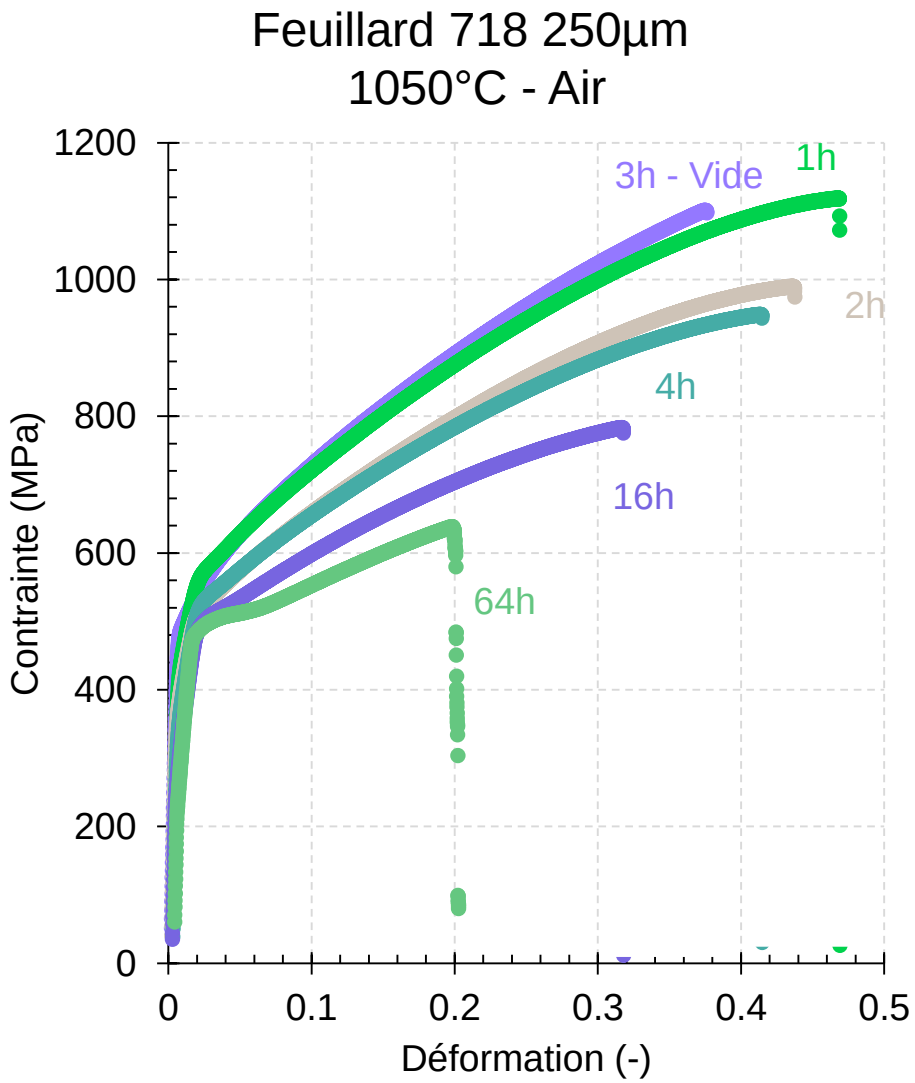




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Développement d'un module getter





Oxydation sur paroi mince ou structure lattice en FA

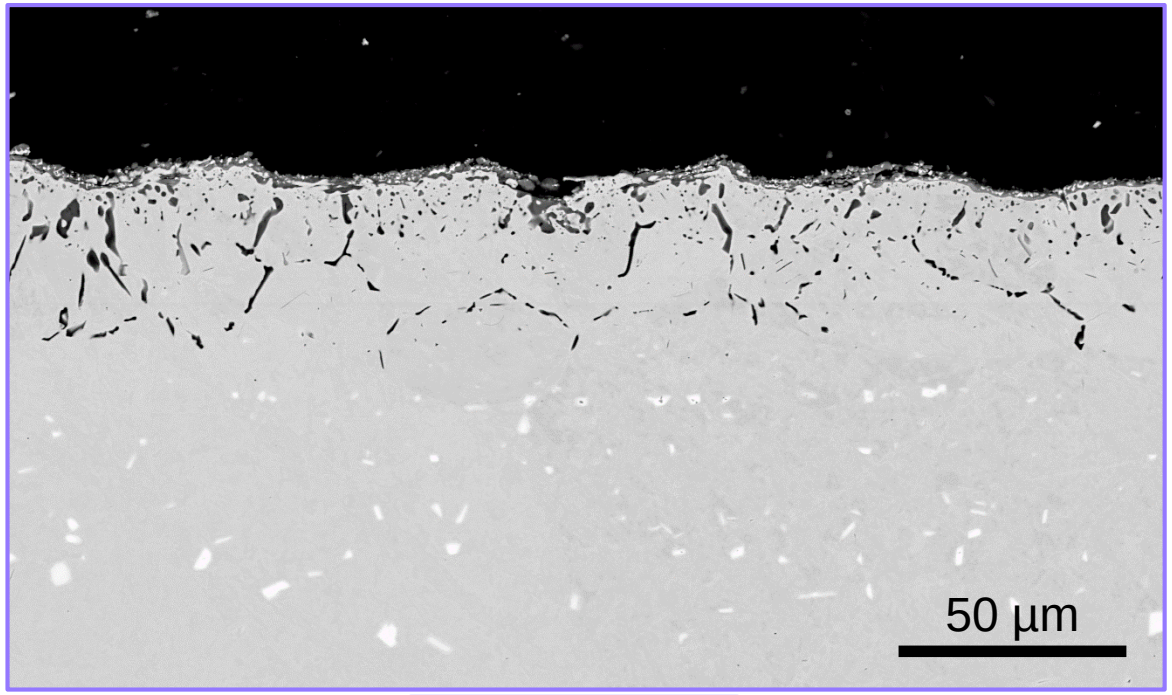


Post-doc A.Prillieux

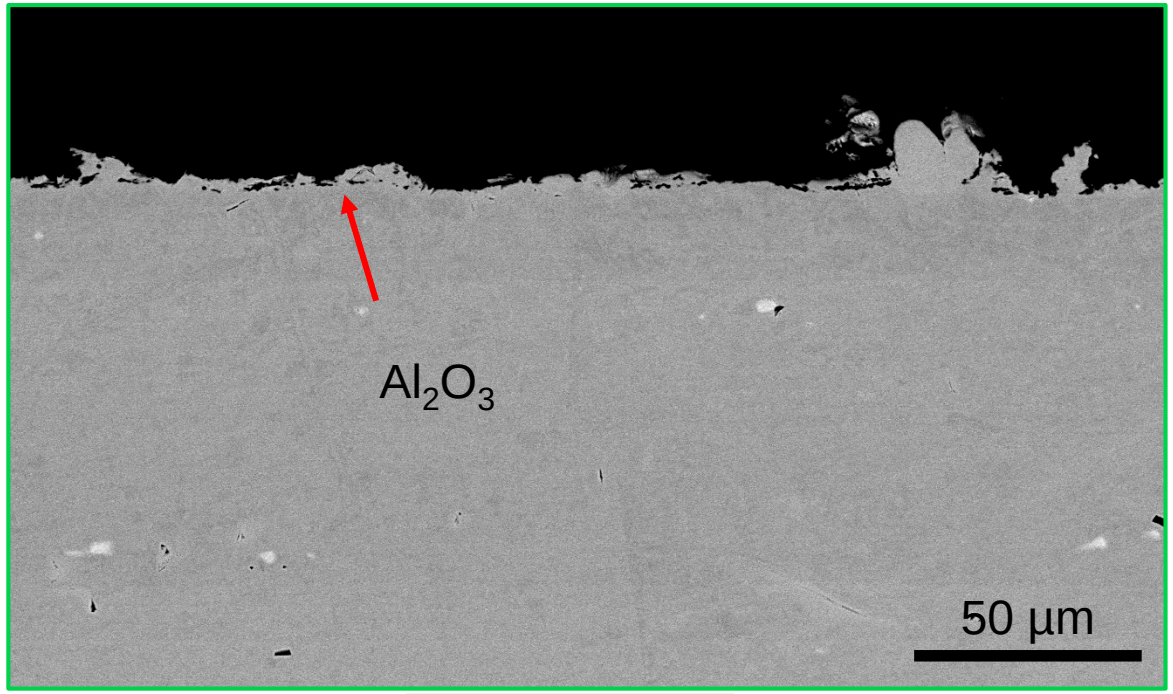
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Développement d'un module getter

Observations MEB, 718 forgé
CIC 1200°C, 1000 bar, 4h, Ar 4.8 **sans** ou **avec** modules getters



Sans Getter



Avec Getter

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Conclusions

La maitrise des atmosphères lors des procédés est primordiale pour assurer les performances des pièces produites

Au cours de la fabrication → **Etude** sur l'évolution de la composition du gaz dans une machine de fabrication ?

- Capteur d'ambiance → Ok
- Au plus près des pièces ?

Au cours des traitements thermiques → **CIC** sur pièces **architecturées** et/ou **non usinables**

Projet HIPPOME

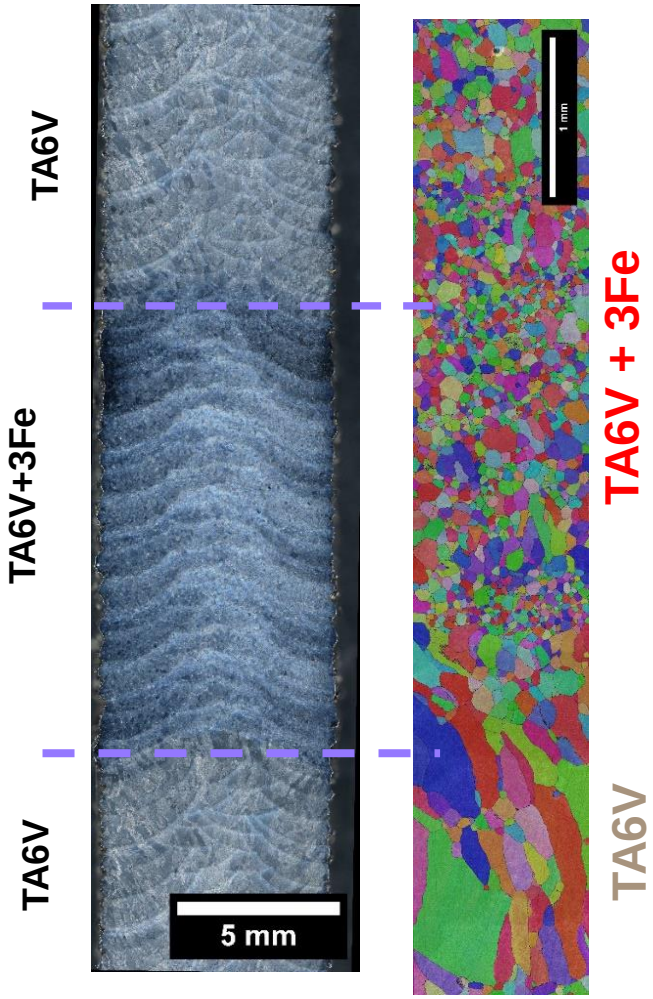
- Compréhension des mécanismes d'oxydation au cours du CIC
- Instrumentation réussie d'un four CIC avec prélèvement de gaz dans le four
- Développement d'une solution technique pour éviter l'oxydation

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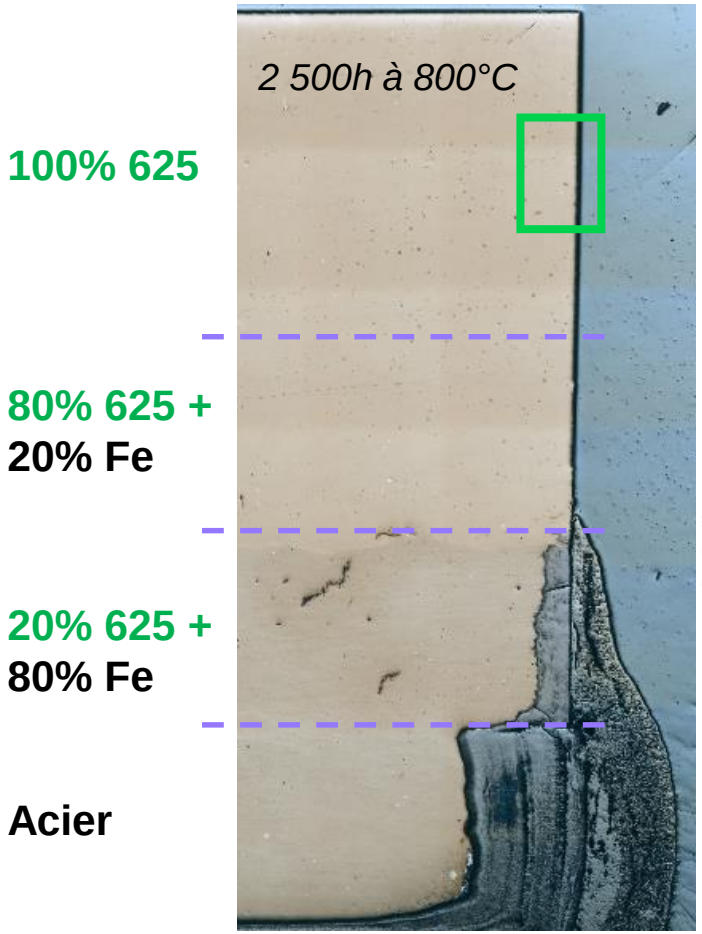
Merci pour votre attention

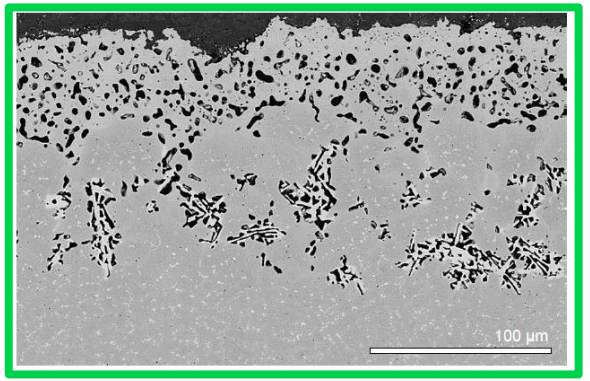
Beaucoup d'autres sujets FA !
N'hésitez pas à venir discuter !

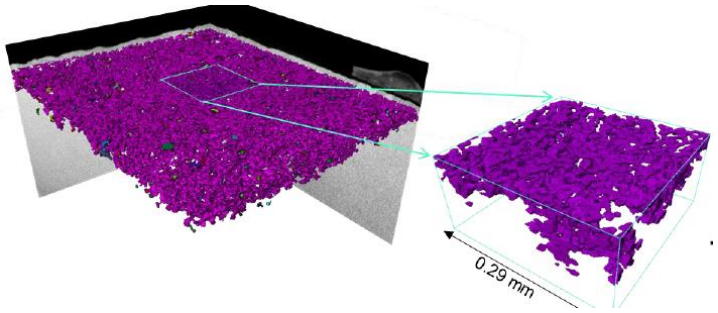
Gradient de composition
effet sur les microstructures



Gradient de composition
corrosion à chaud







[8] Agathe C., Thèse, 2024

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