High Temperature Corrosion of Nickel-Based Superalloys in an Oxy-Fuel Gas Environment

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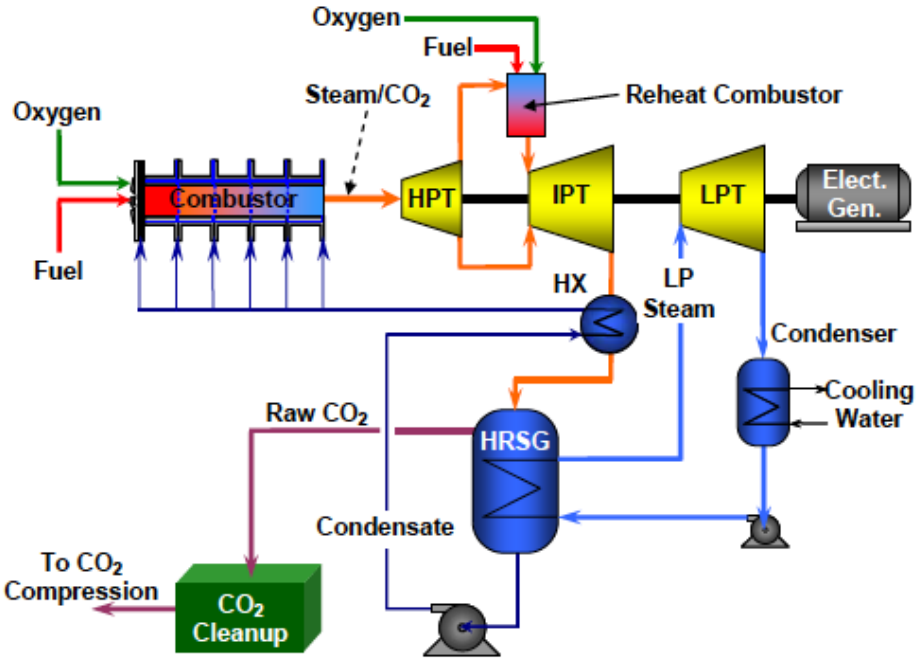
CHALMERS
UNIVERSITY OF TECHNOLOGY

The High Temperature Corrosion Centre

SIEMENS

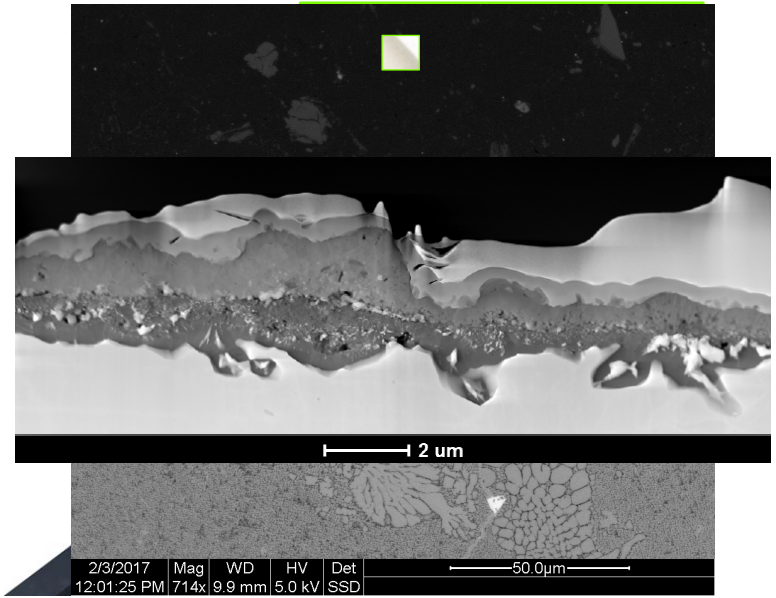
Why Oxy-fuel combustion?

- carbon capture and storage (CCS) system to mitigate carbon dioxide (CO_2) emissions.
- substantially reduced NO_x emissions
- combustion products can be reduced to mainly steam (H_2O) and CO_2.



Project aims

- Describe oxy-fuel corrosion conditions with high water vapour and carbon dioxide levels
- Find underlying corrosion mechanisms during high temperature exposures in these environments
- Suggest guidelines for selecting materials with the ability to resist corrosion in these environment



Materials and Methods

- **Exposure environment: 90% CO_2- 10% H_2O at 900 °C**
- **Exposure duration: 270 h, 532 h and 1056 h**
- **In order to simulate a service environment, temperature cycles of 65 hours, was used.**
- **Samples:**
  1. **CM247: poly crystal**

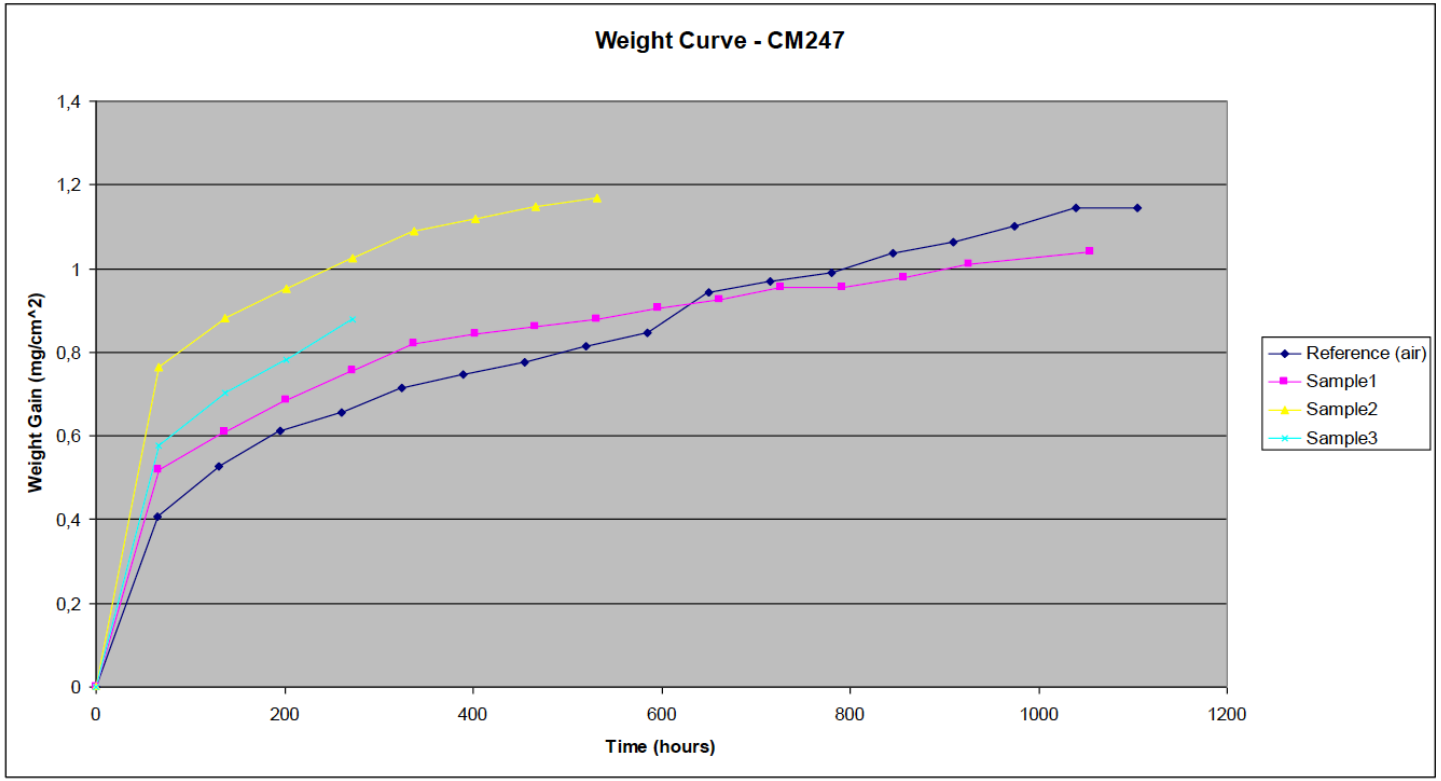
wt.%	Al	B	C	Co	Cr	Hf	Mo	Ni	Ta	Ti	Zr	V	W
min	5,40	0,01	0,07	9,00	8,00	1,40	0,40		3,10	0,60	0,005		9,30
max	5,70	0,02	0,08	9,50	8,50	1,60	0,60	rem	3,30	0,90	0,02	0,10	9,70

  2. **STAL 15: single crystal**

wt.%	Al	C	Si	Ce	Co	Cr	Hf	Mo	Ni	Ta	Ti	W
min	4,50	0,010	0,23	0,0005	5,00	14,80	0,08	0,90		7,80		3,80
max	4,70	0,020	0,27	0,005	5,40	15,20	0,12	1,10	rem	8,20	0,1	4,20

  3. **IN792**
  4. **IN939**

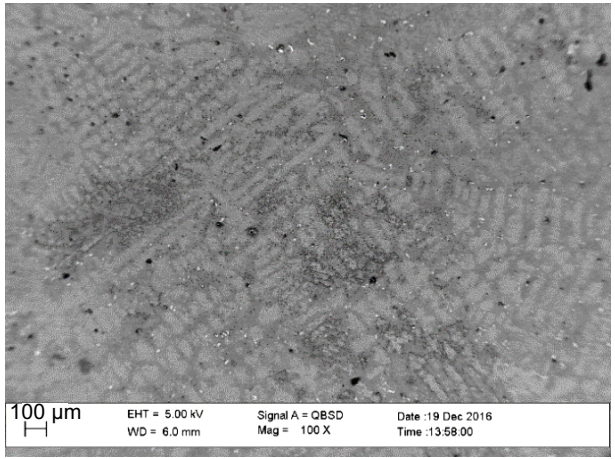
Mass Gain



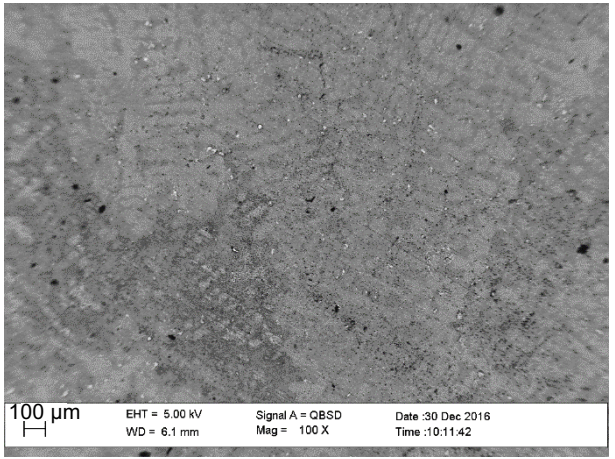
CM247

Top View SEM BSE

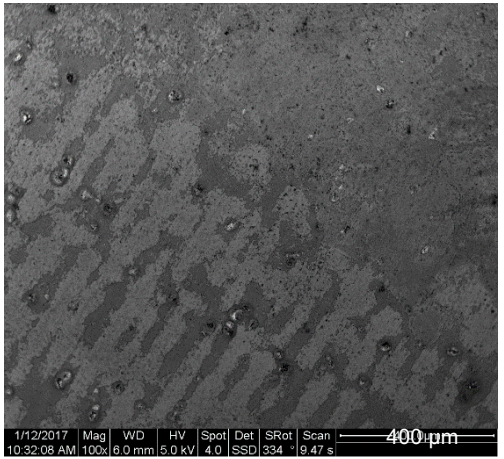
270h



532h

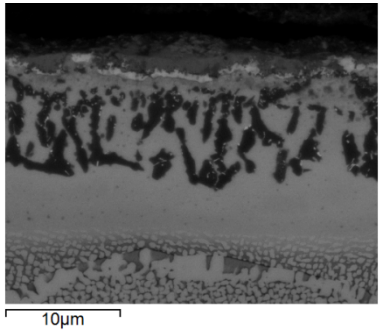


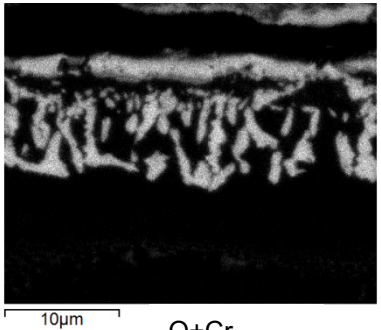
1056h



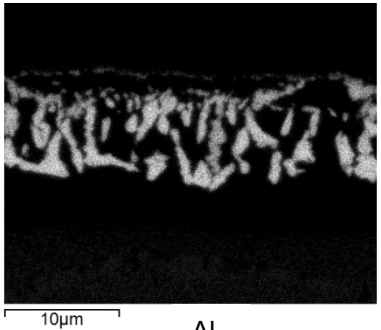
CM247

Thick oxide region

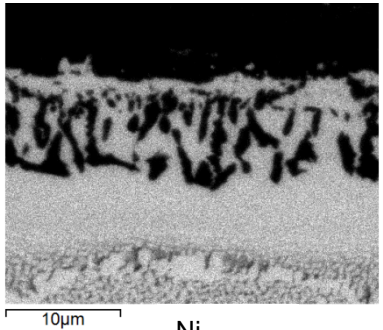




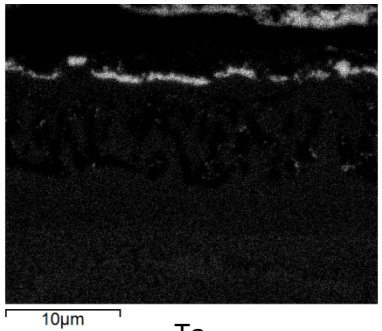
O+Cr



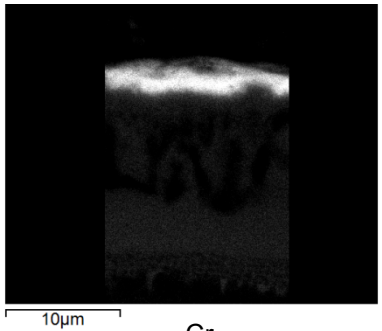
Al



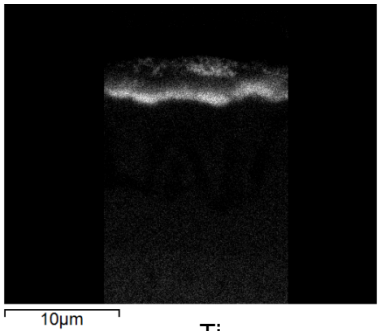
Ni



Ta



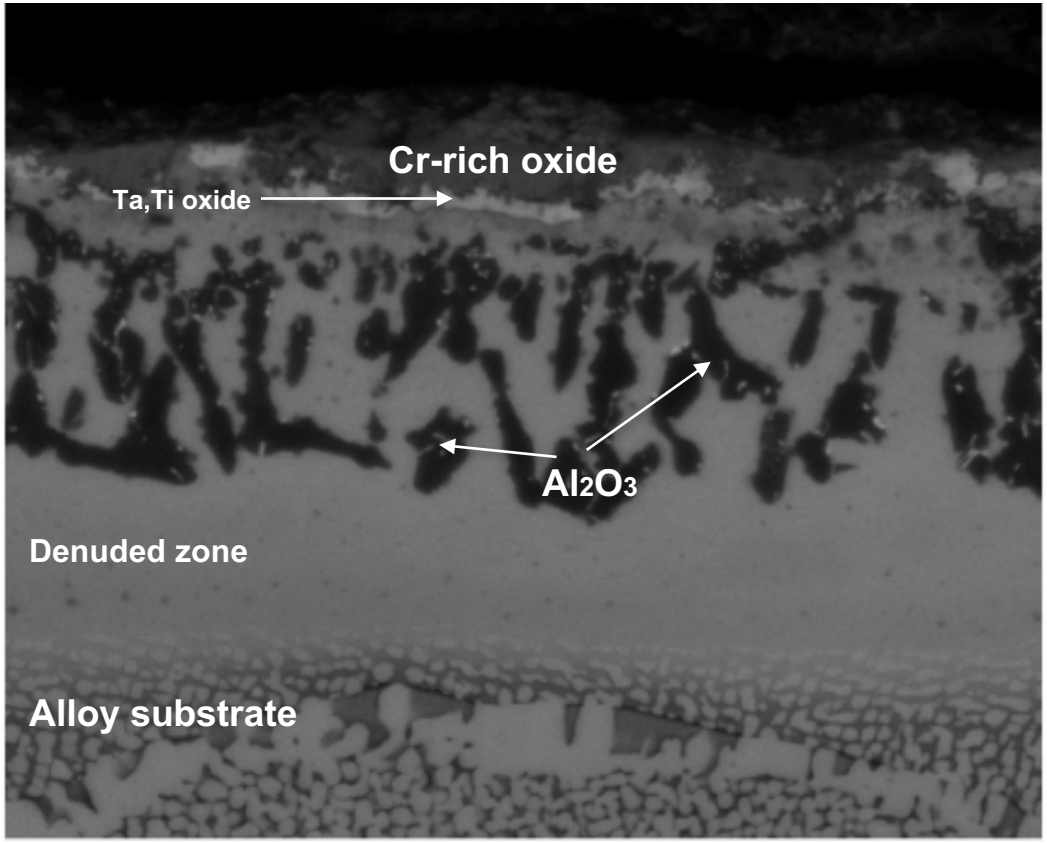
Cr



Ti

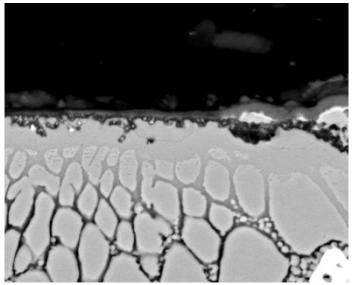
CM247-270 h

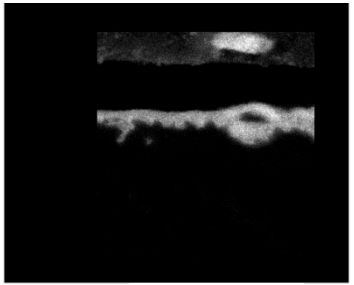
Thick oxide cross-section summary



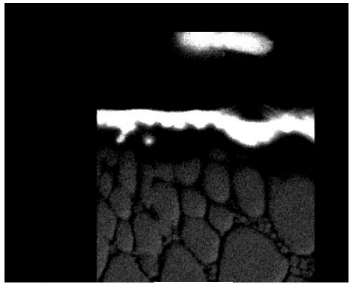
CM247-270 h

Thin oxide region

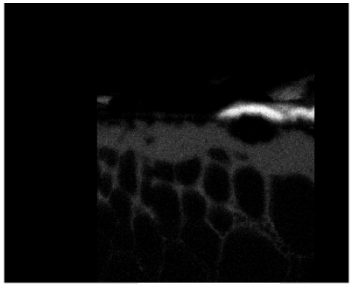




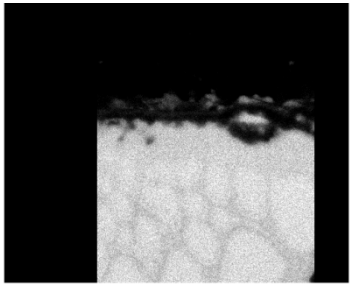
O



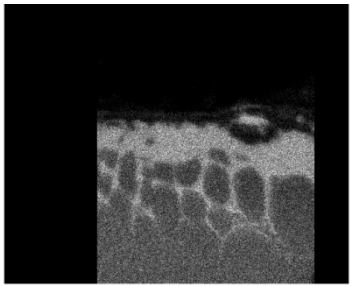
Al



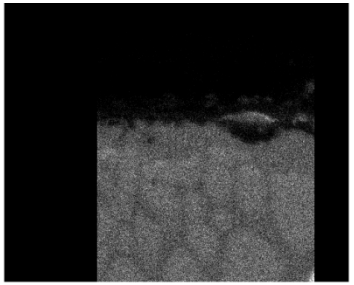
Cr



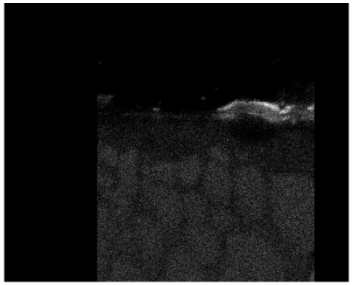
Ni



Co



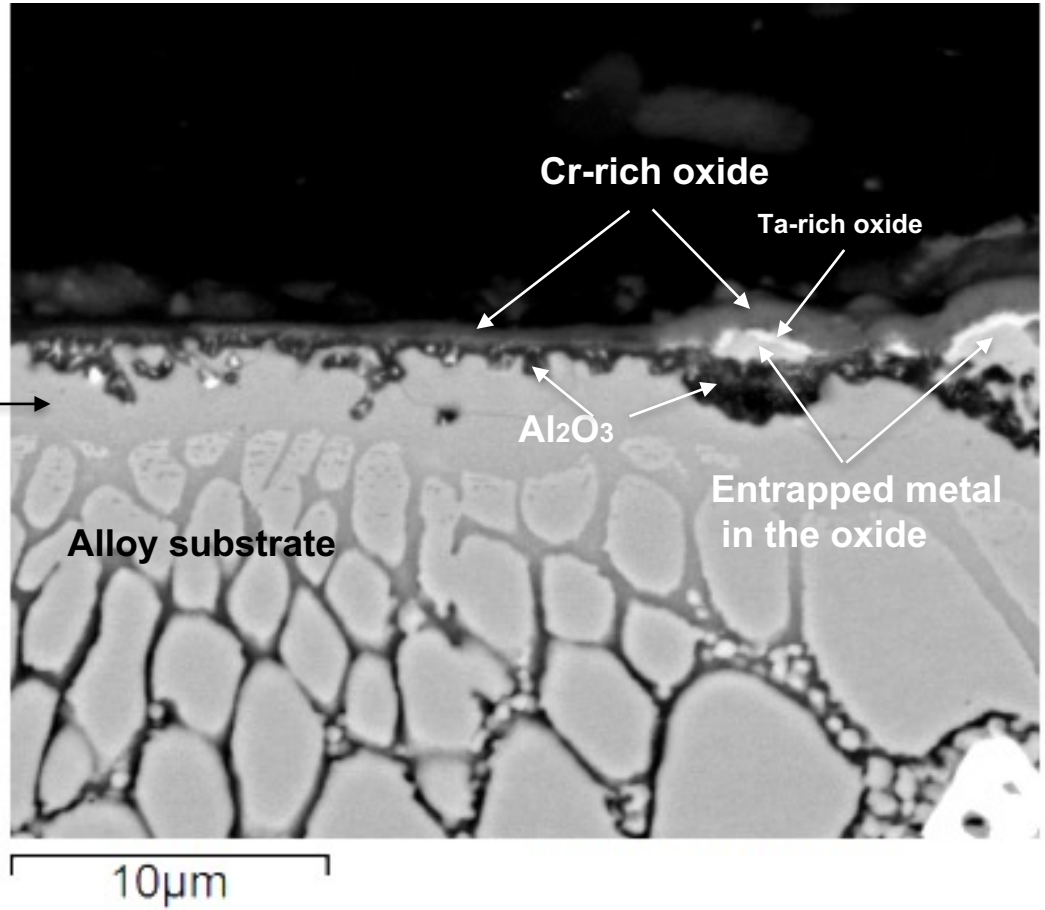
Ta



Ti

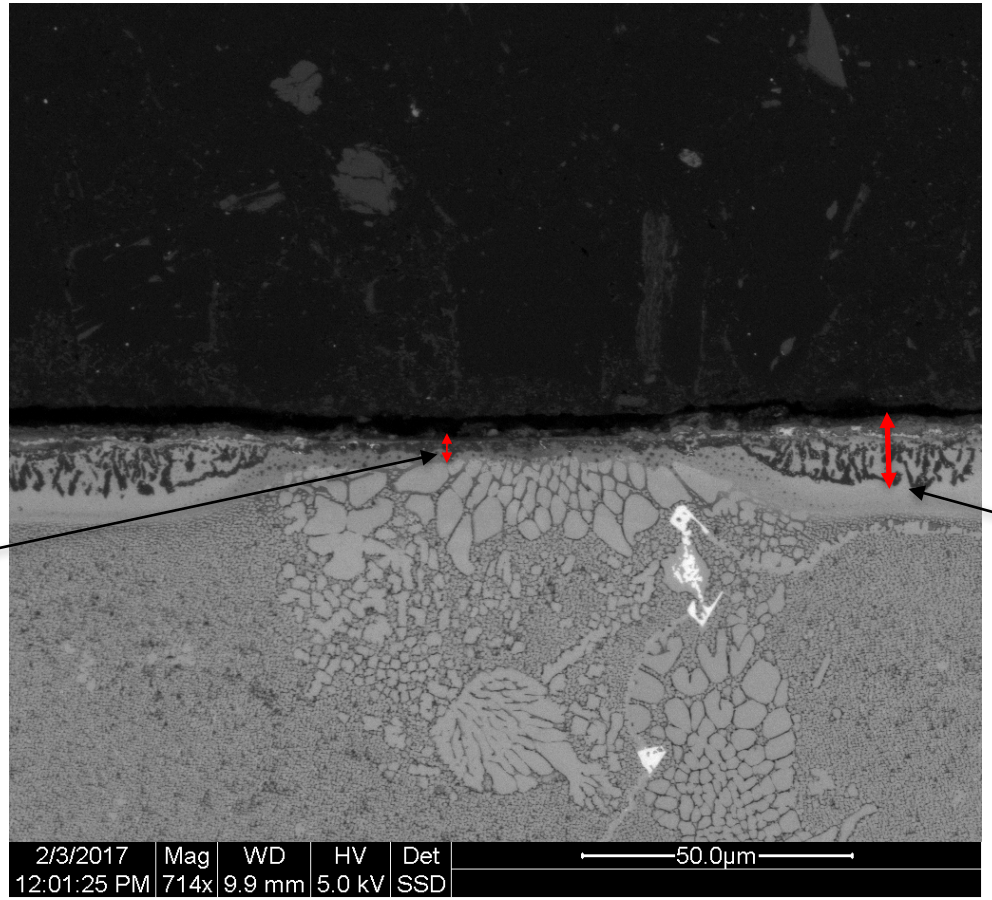
CM247-270 h

Thin oxide cross-section summary



CM247-270 h

Cross-section SEM BSE

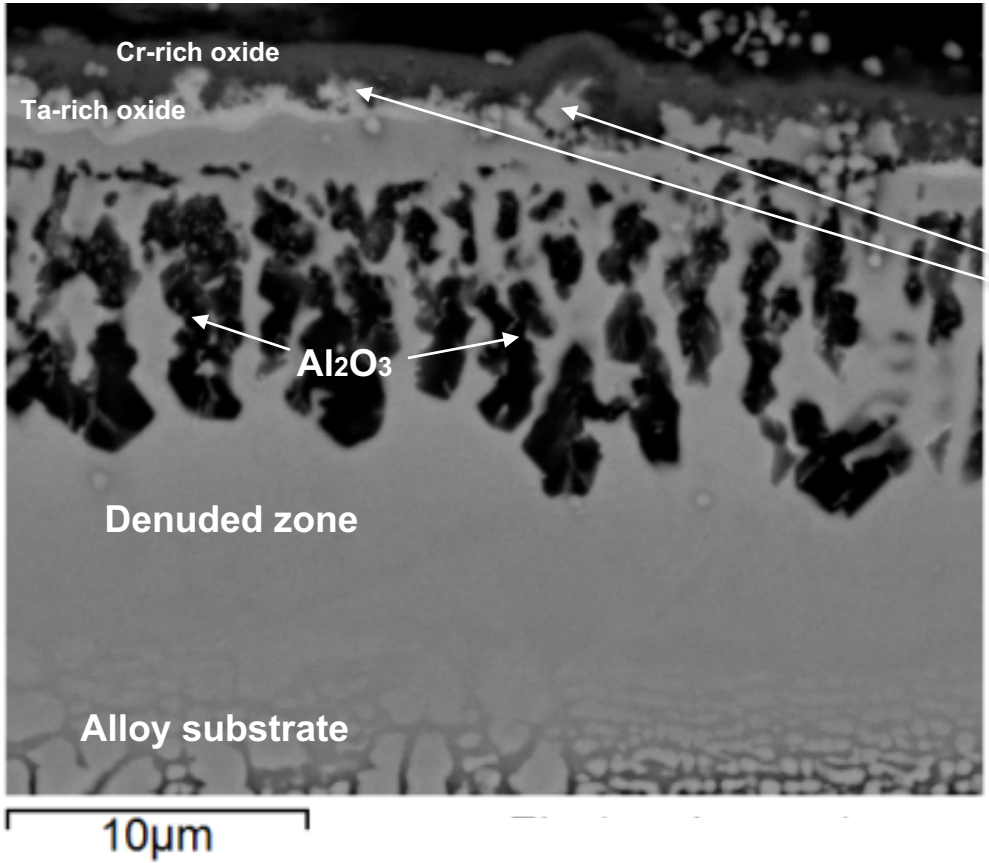


Thin oxide ≈ 2-3 µm

Thick oxide ≈ 13 µm

CM247-270 h

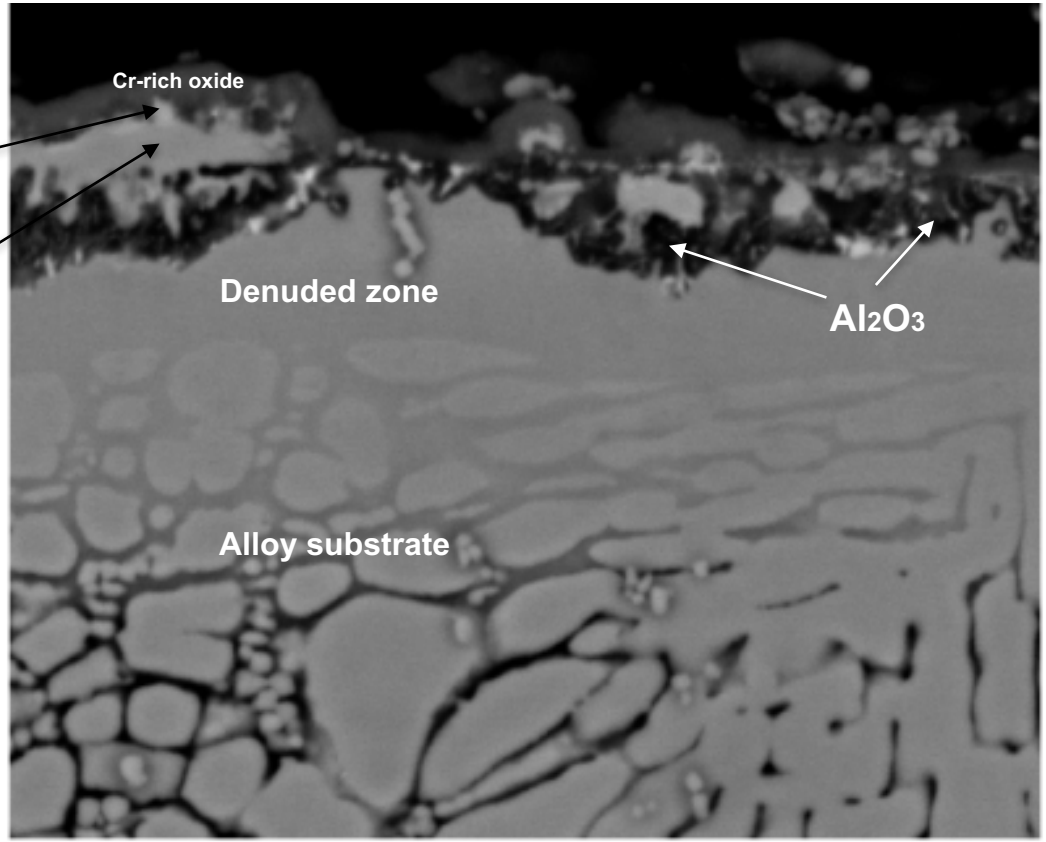
Thick oxide cross-section summary



Entrapped metal in the oxide

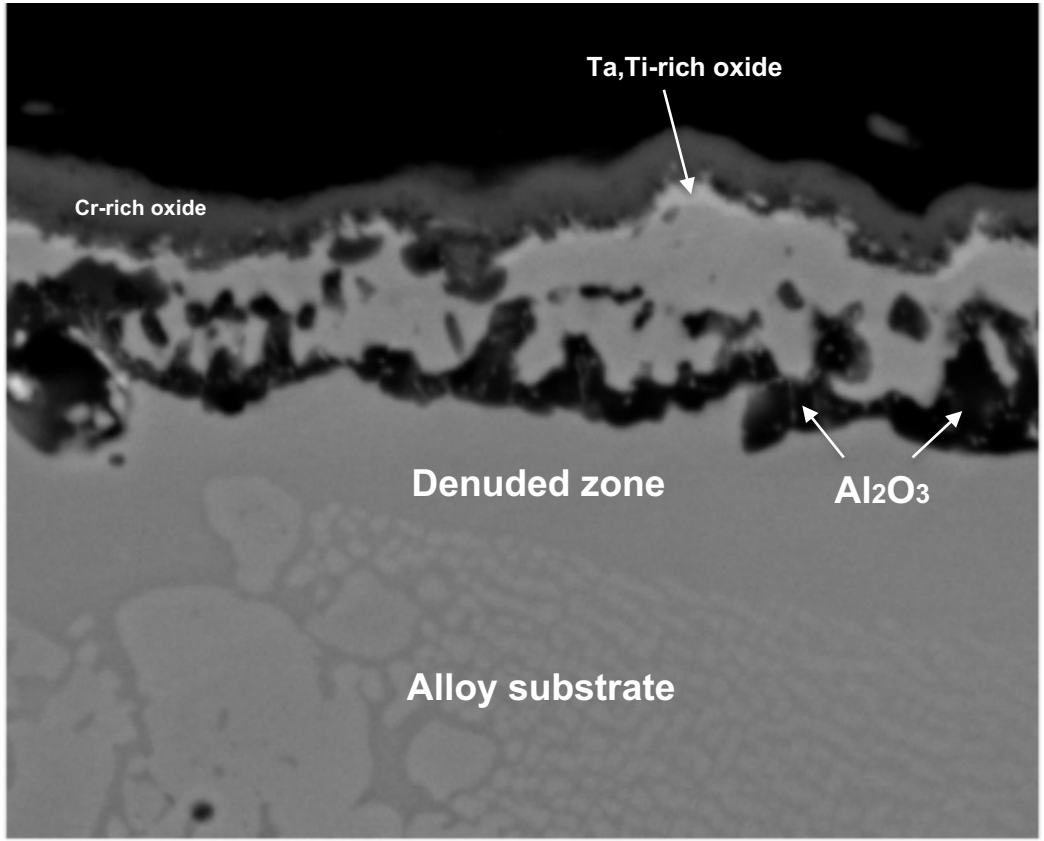
CM247-532 h

Thin oxide cross-section summary



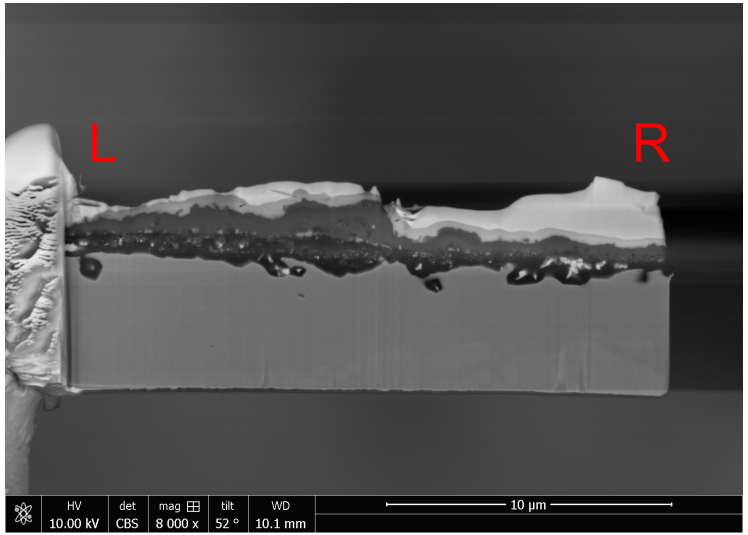
CM247-532 h

Thick oxide cross-section summary



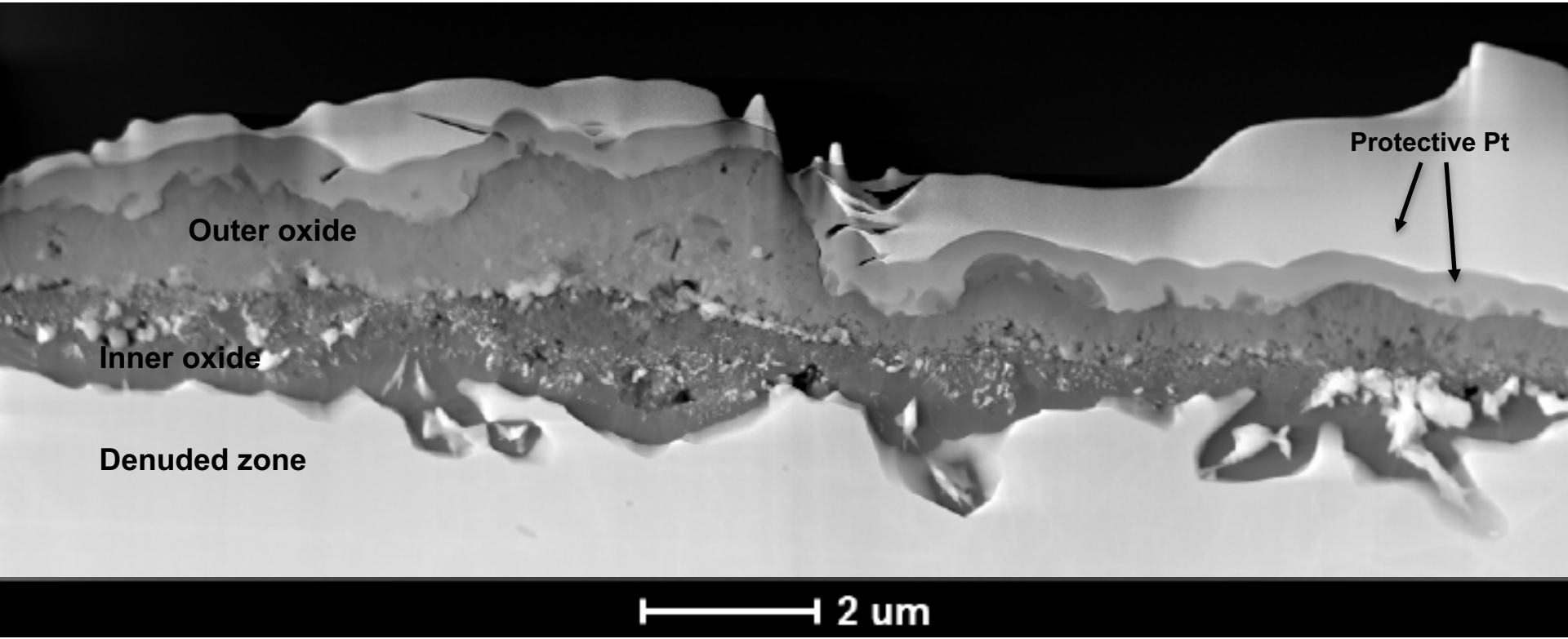
CM247-1056 h

FIB/SEM cross-section lift-out
Thin oxide



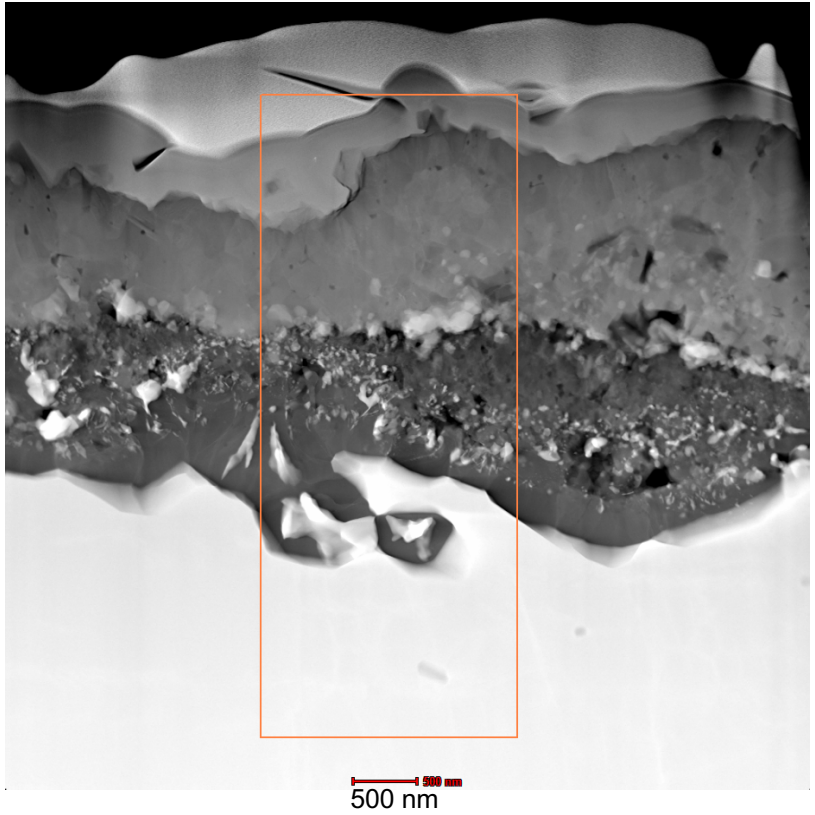
CM247-1056 h

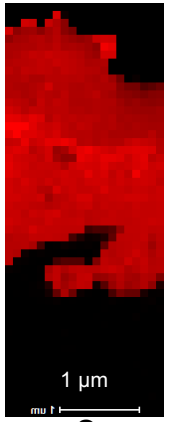
STEM cross-section

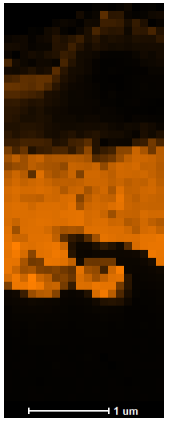


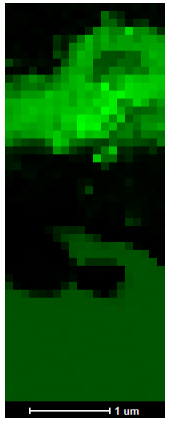
CM247-1056 h

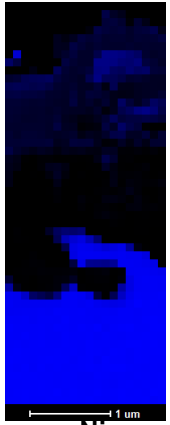
STEM/EDX cross-section

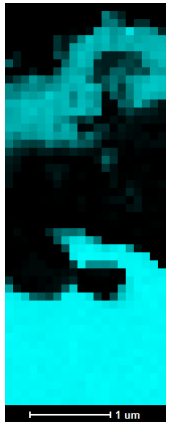


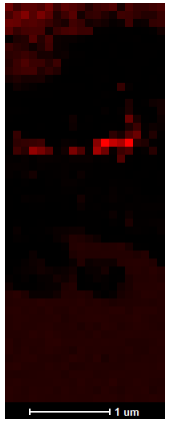


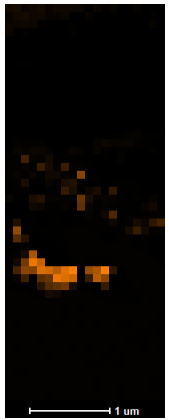






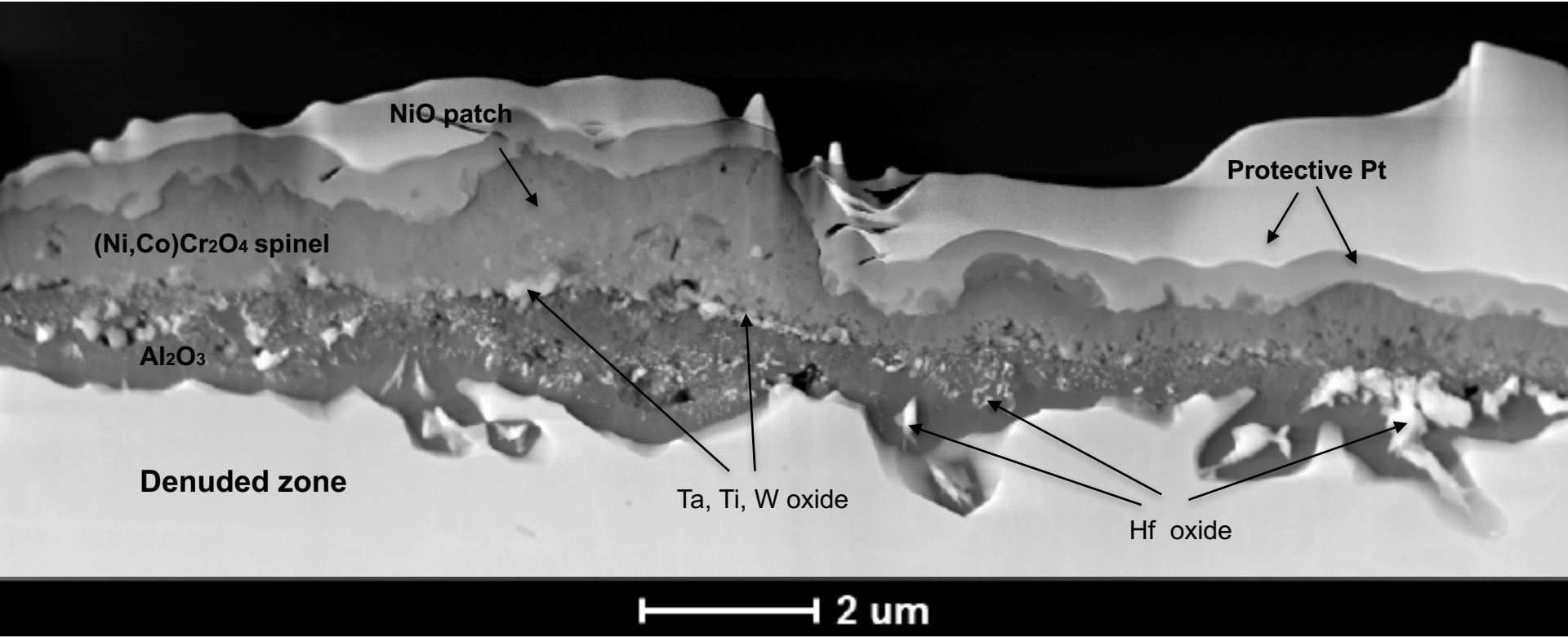






CM247-1056 h

STEM cross-section summary



CM247-1056 h

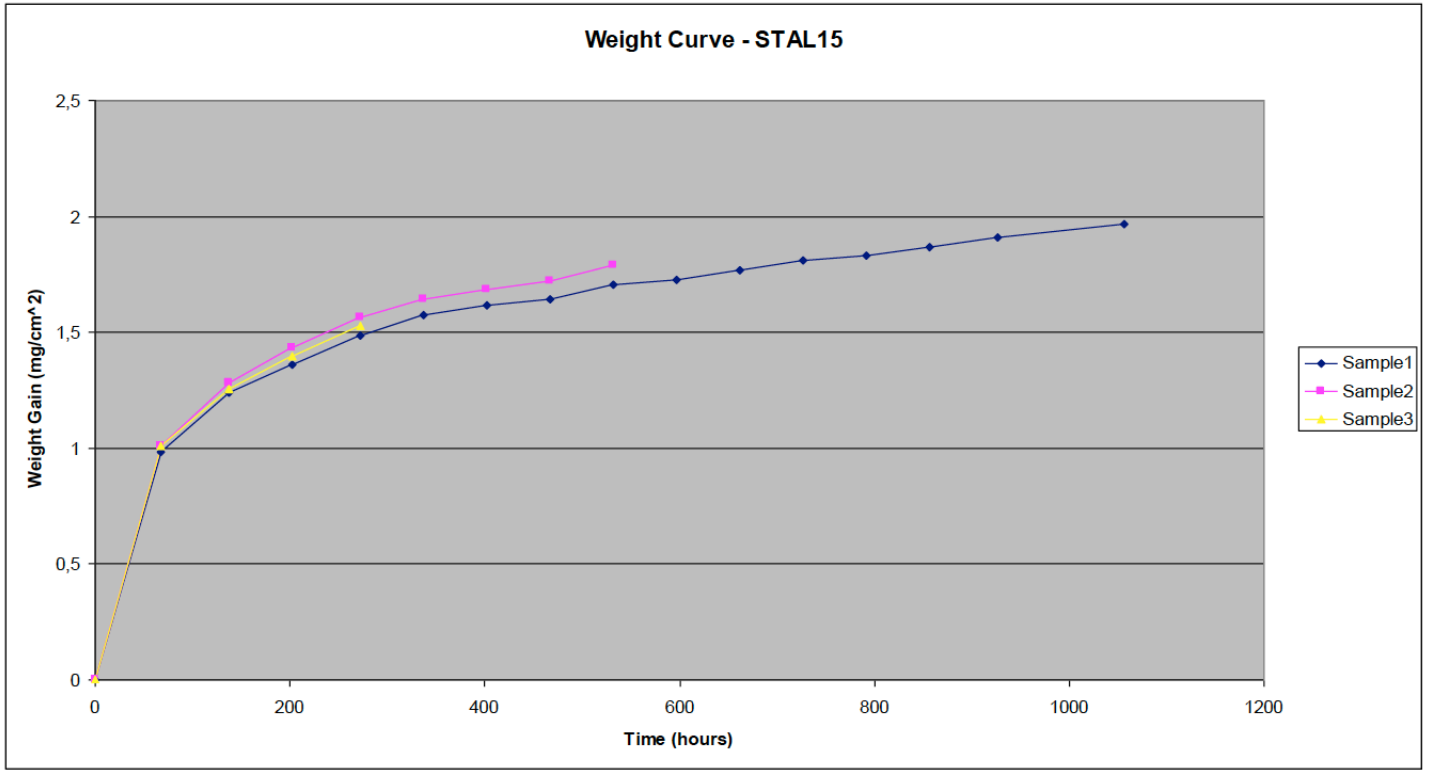
Summary of CM247

- Thin and thick oxide regions
- Total oxide thickness increases about 2μm
- STEM/EDX suggests the top layer is likely $(Ni,Co)Cr_2O_4$ spinel
- After 1056 h exposure the Al_2O_3 internal oxidation is interconnected providing good barrier against further oxidation

STAL 15 single crystal

wt.%	Al	C	Si	Ce	Co	Cr	Hf	Mo	Ni	Ta	Ti	W
min	4,50	0,010	0,23	0,0005	5,00	14,80	0,08	0,90		7,80		3,80
max	4,70	0,020	0,27	0,005	5,40	15,20	0,12	1,10	rem	8,20	0,1	4,20

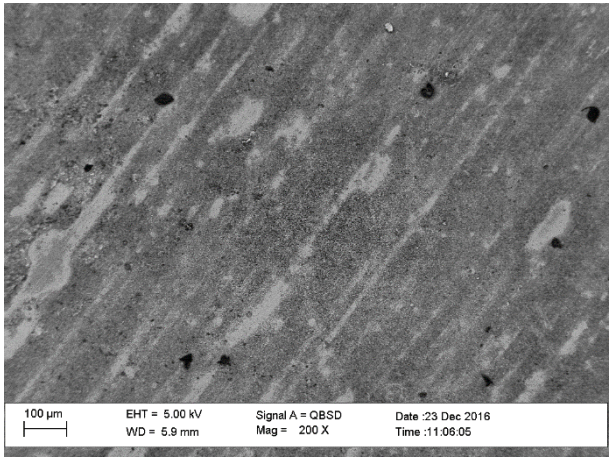
Mass Gain



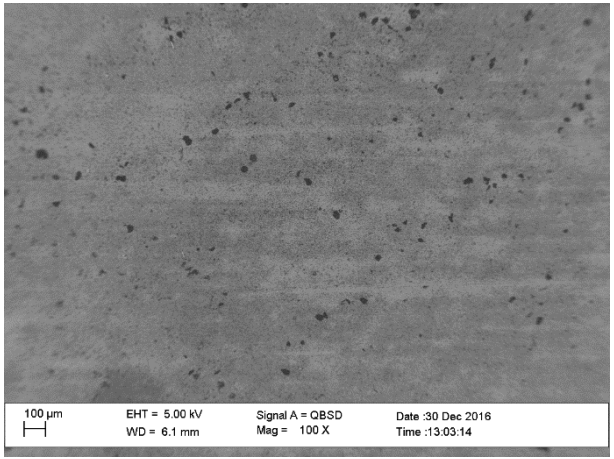
STAL 15

Top View SEM BSE

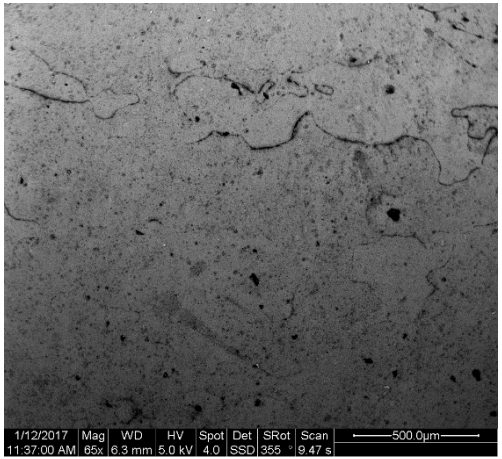
270h



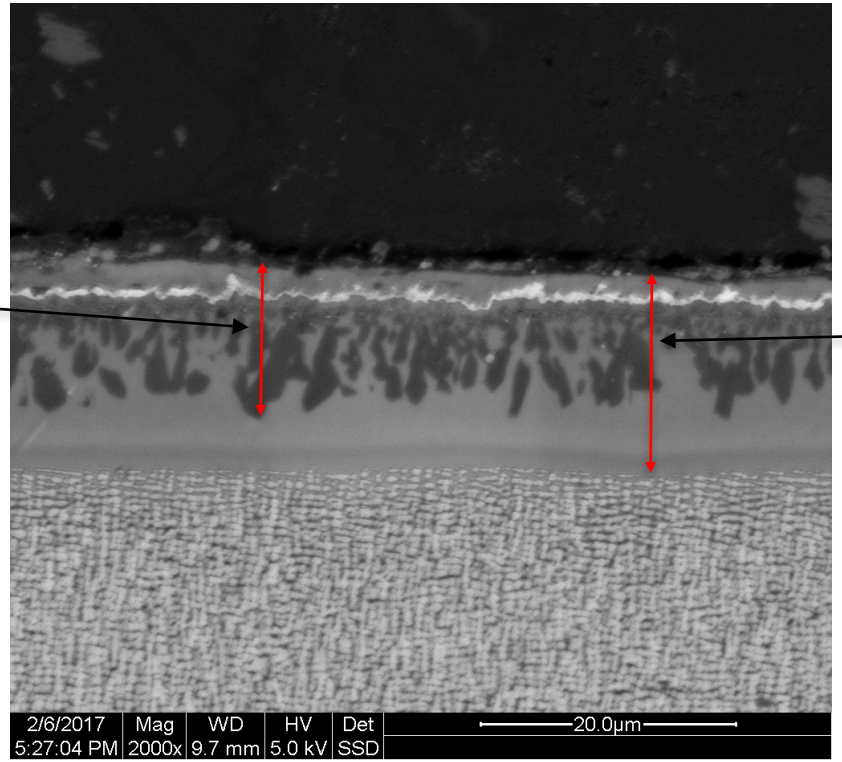
532h



1056h

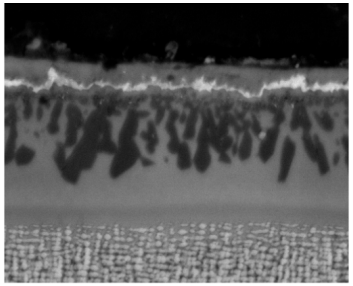


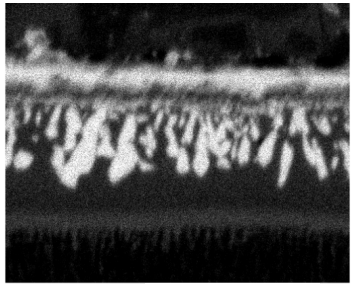
Cross-section SEM BSE



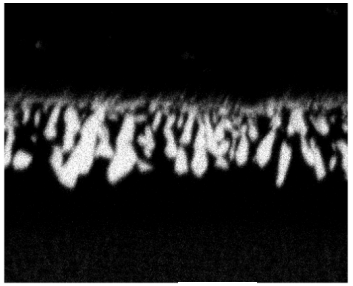
STAL 15 - 270 h

Cross-section EDX elemental map

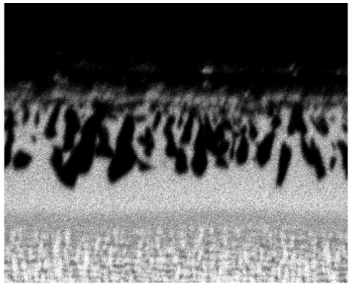




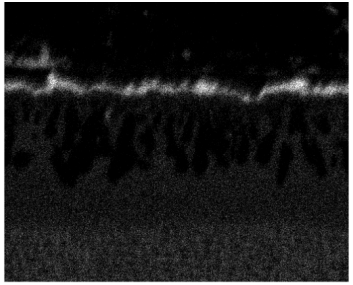
O+Cr



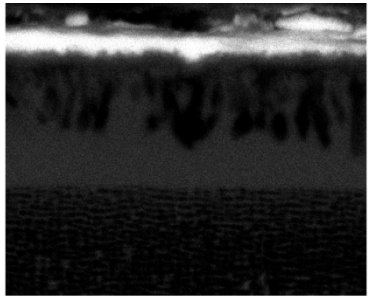
Al



Ni



Ta

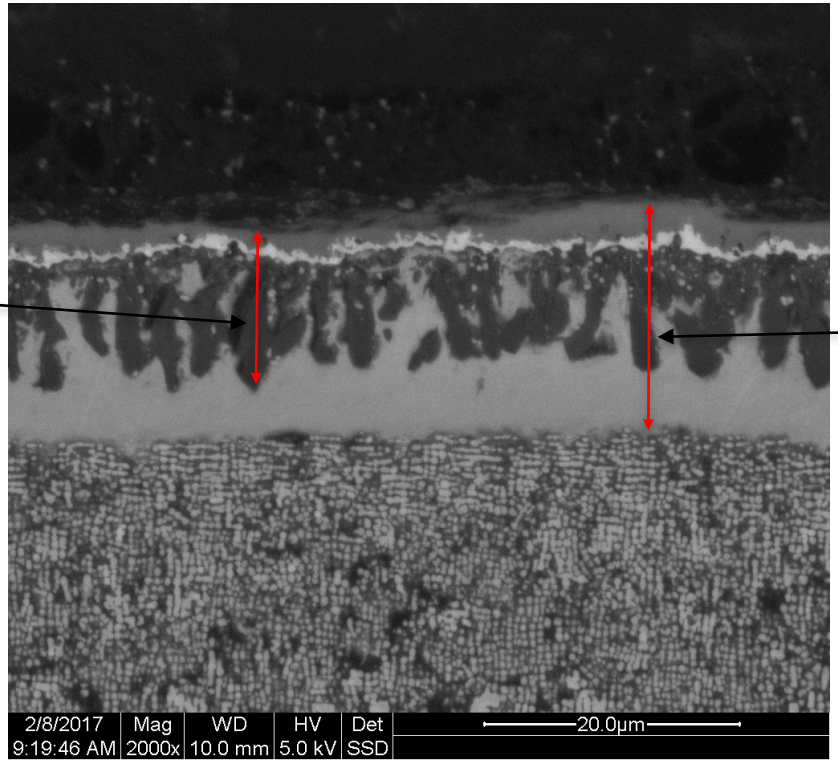


Cr

STAL 15 – 270 h

Cross-section SEM BSE

≈ 13-14 µm

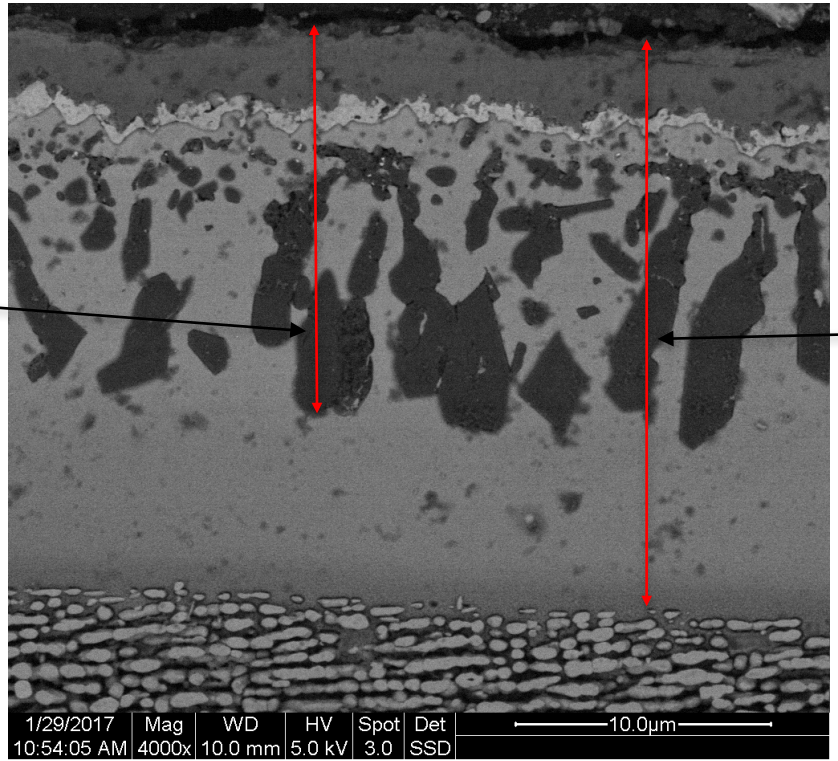


≈ 18 µm

STAL 15 – 532 h

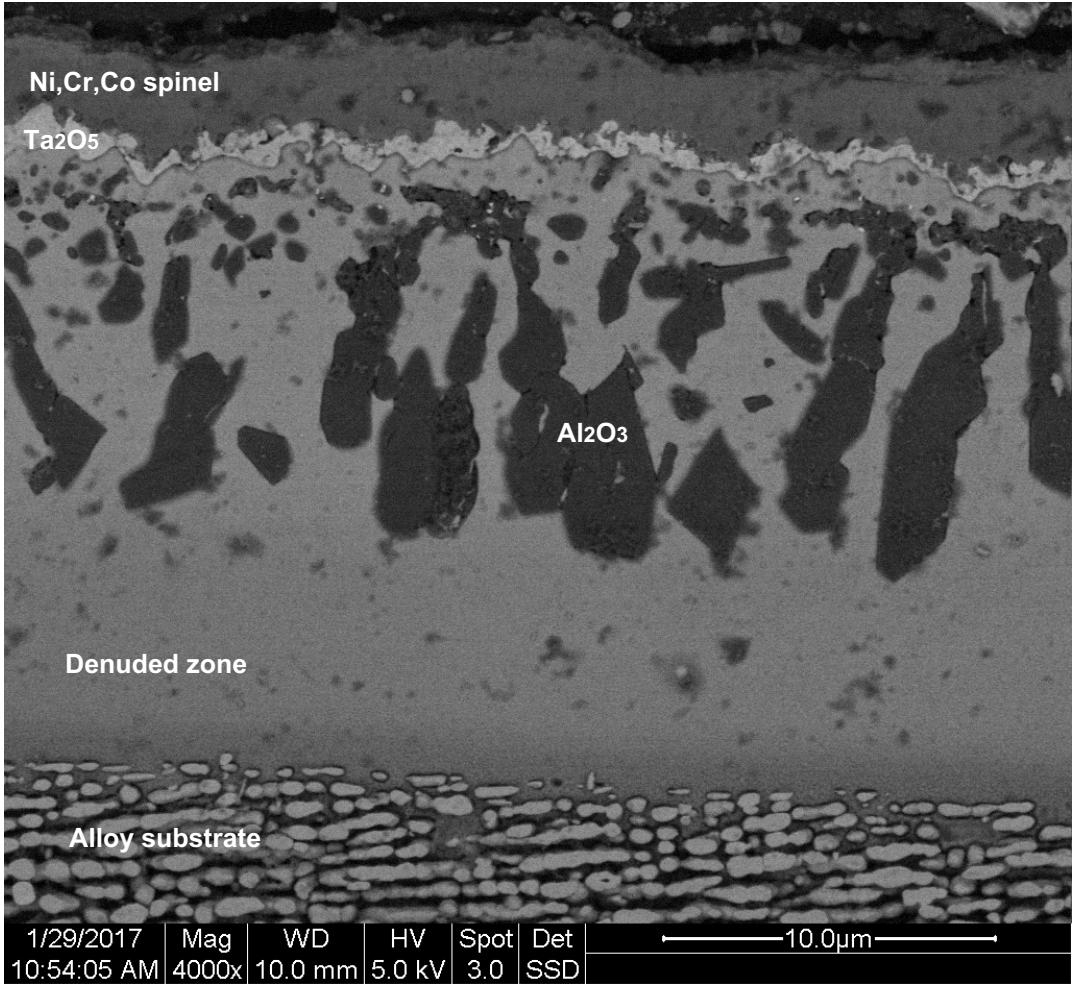
Cross-section SEM BSE

≈ 14-16 µm



≈ 22 µm

STAL 15 – 1056 h

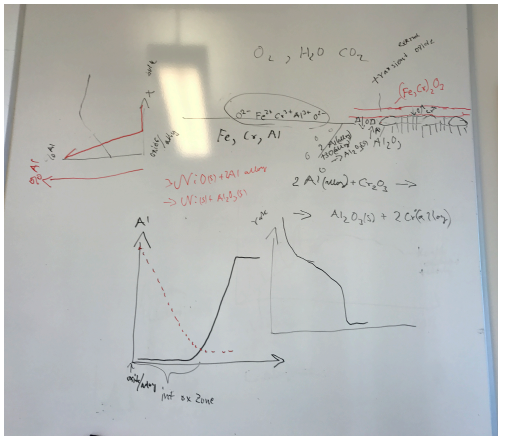

Ni,Cr,Co spinel
Ta2O5
Al2O3
Denuded zone
Alloy substrate
1/29/2017
10:54:05 AM
Mag
4000x
WD
10.0 mm
HV
5.0 kV
Spot
3.0
Det
SSD
10.0µm

Summary of STAL 15

- From top view images there seems to be some spallation
- Uniform oxide
- The oxide thickness increases from ≈ 12 μm after 270 h to ≈16 μm after 1056 h
- The denuded area increases from ≈ 4 μm to ≈ 6 μm below the oxide
- The top layer could be $NiCr_2O_4$ spinel
- Surprisingly bad oxidation behaviour in the investigated environment; no protective Al_2O_3 has formed

Future work

- TEM investigation of STAL 15 after 1056 h
- SEM/EDX analysis of IN792 and IN939
- TEM/EDX analysis of IN792 and IN939 after 1056 h
- Understanding the underlying mechanism(s)



Thank you for your attention