

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

Mohammad Sattari*, Mats Halvarsson*, Lars-Gunnar Johansson*,
Marie-Aude Porter**, Helena Oskarsson**

* Chalmers University of Technology

** Siemens Industrial Turbomachinery AB

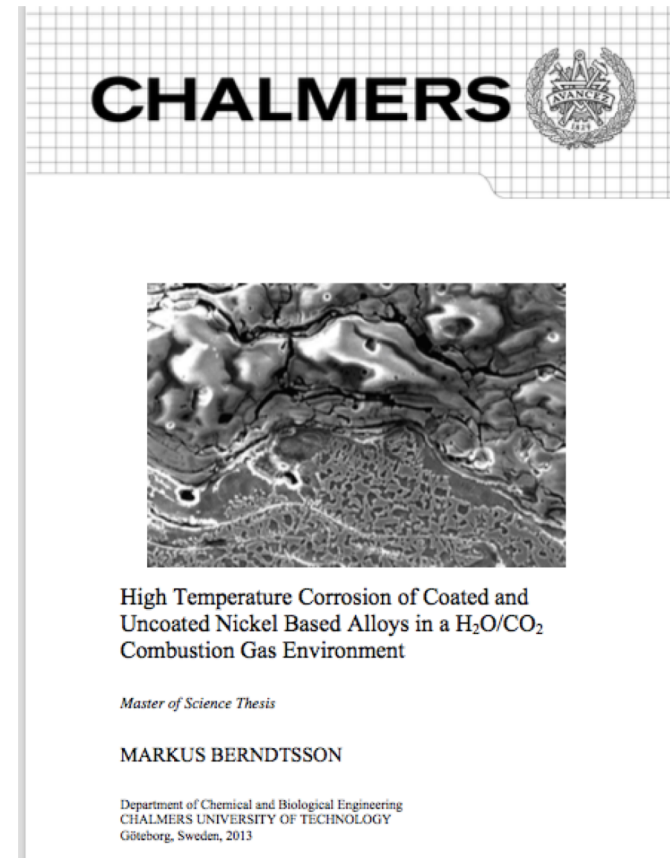


SIEMENS



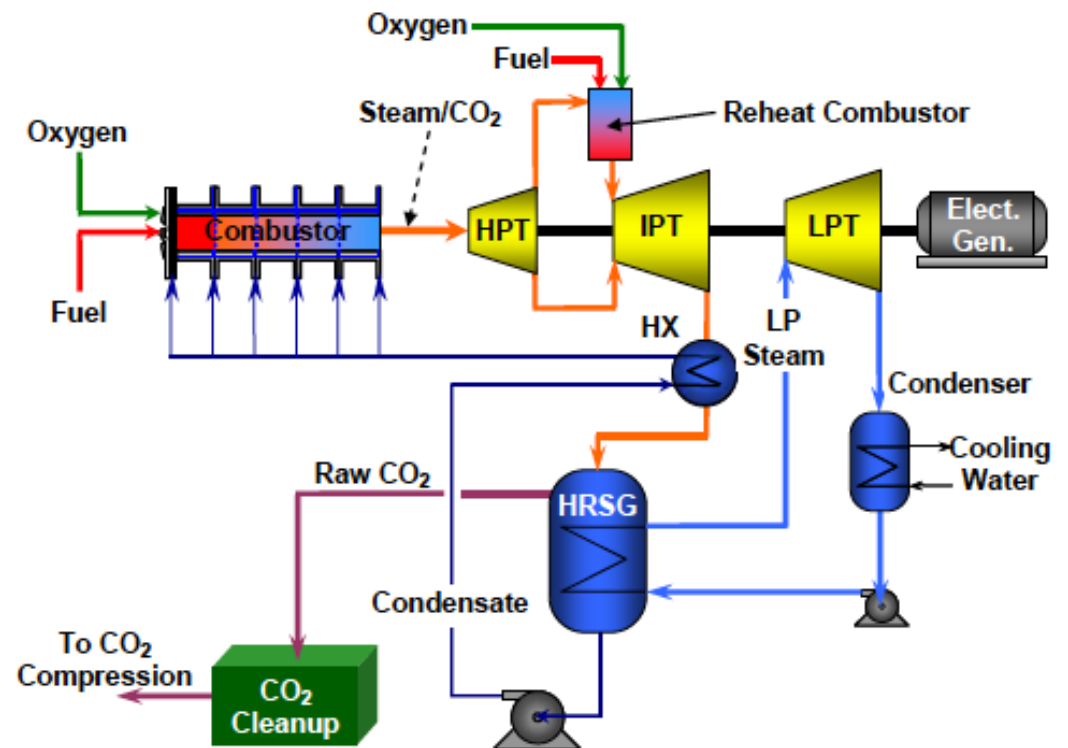
Project initiation

- Master thesis project:
Markus Berndtsson
- Supervisor:
Lars-Gunnar Johansson



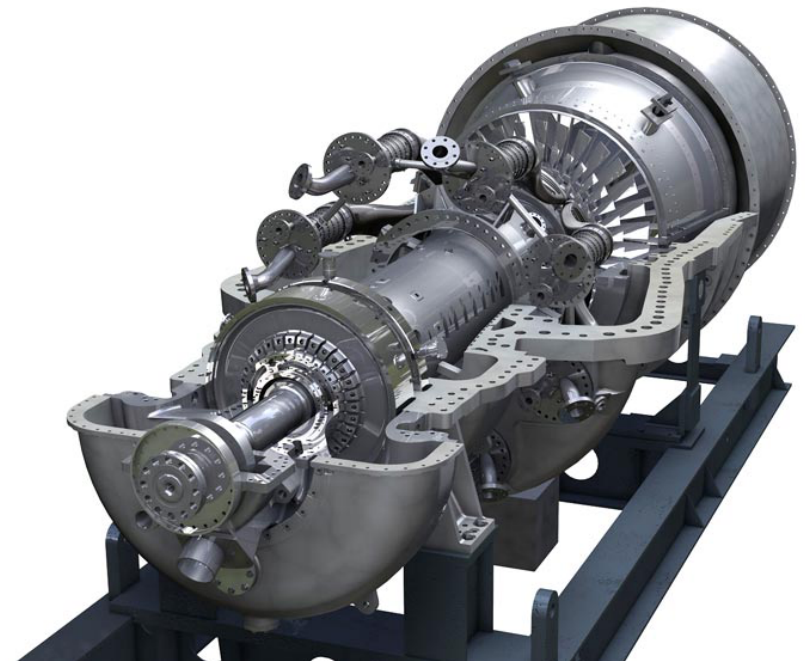
Why oxy-fuel combustion?

- 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



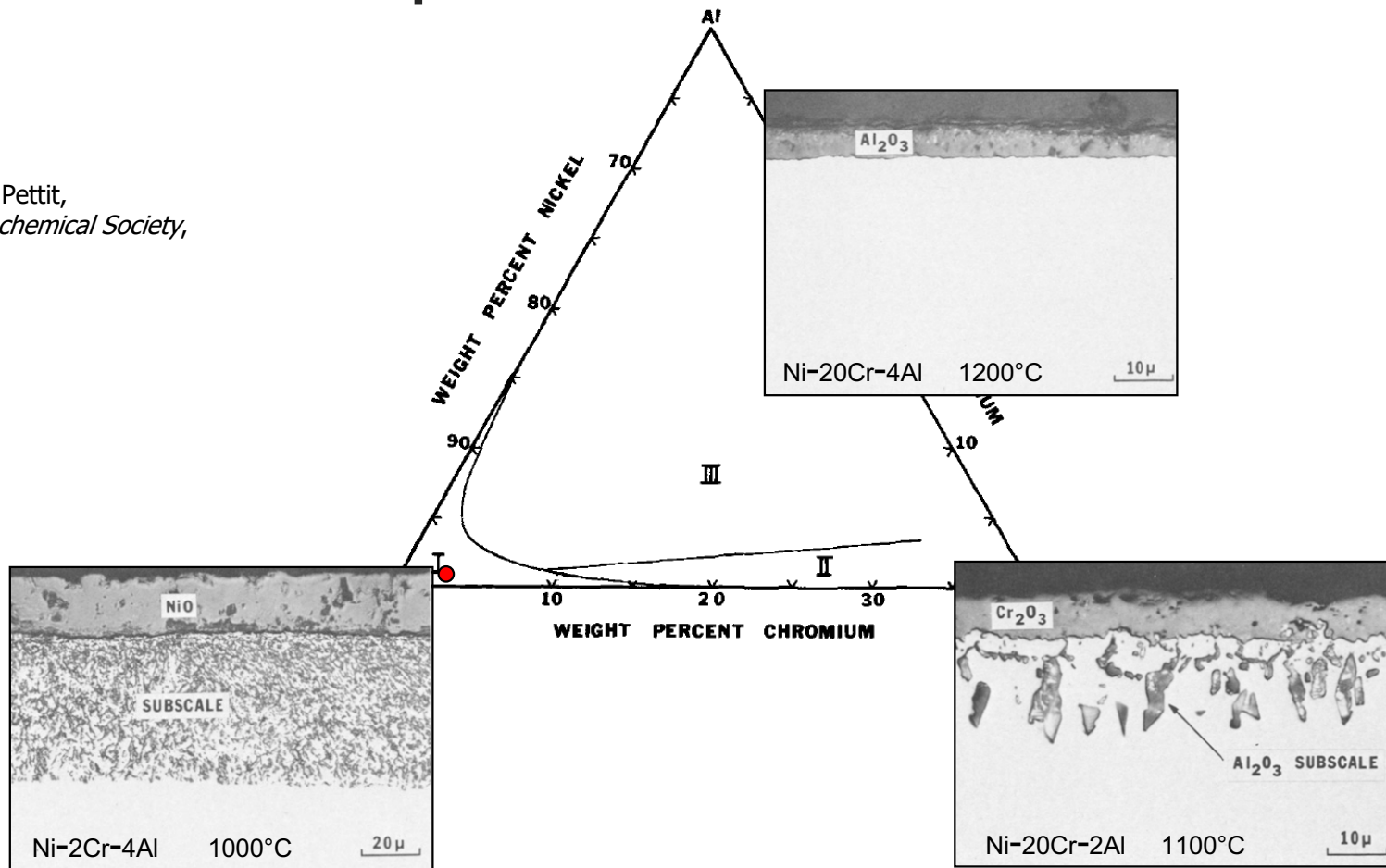
Materials

	Al	B	Si	C	Co	Cr	Hf	Mo	Nb	Ta	Ti	Zr	V	Ce	W
CM247	5.40-	0.01-	-	0.07-	9.00-	8.0-	1.40-	0.40-	-	3.10-	0.60-	0.005-	≤0.	-	9.30
	5.70	0.02		0.08	9.50	8.0	1.60	0.60		3.30	0.90	0.02	1		- 9.70
STAL 15 (Single crystal)	4.50-	-	0.23-	0.010	5.00-	14.8-	0.08-	0.90-	-	7.80-	≤0.1	-	-	0.0005-	3.80
	4.70		0.27	- 0.020	5.40	15.2	0.12	1.10		8.20				0.005	- 4.20
IN792	3.15-	0.01-	-	0.06-	8.50-	12.0-	-	1.65-	-	3.85-	3.75-	0.010-	-	-	3.85
	3.60	0.02		0.10	9.50	13.0		2.15		4.50	4.20	0.025			- 4.50
IN939	1.8-	0.004-	-	0.13-	18.5-	22.0-	-	-	0.9-	1.3-	3.6-	0.02-	-	-	1.8-
	2.0	0.006		0.17	19.5	22.8			1.1	1.5	3.8	0.03			2.2

mass percent

Oxidation map: Ni-Cr-Al at 1000-1200°C

C.S. Giggins and F.S. Pettit,
Journal of the Electrochemical Society,
118, 1782 (1971)



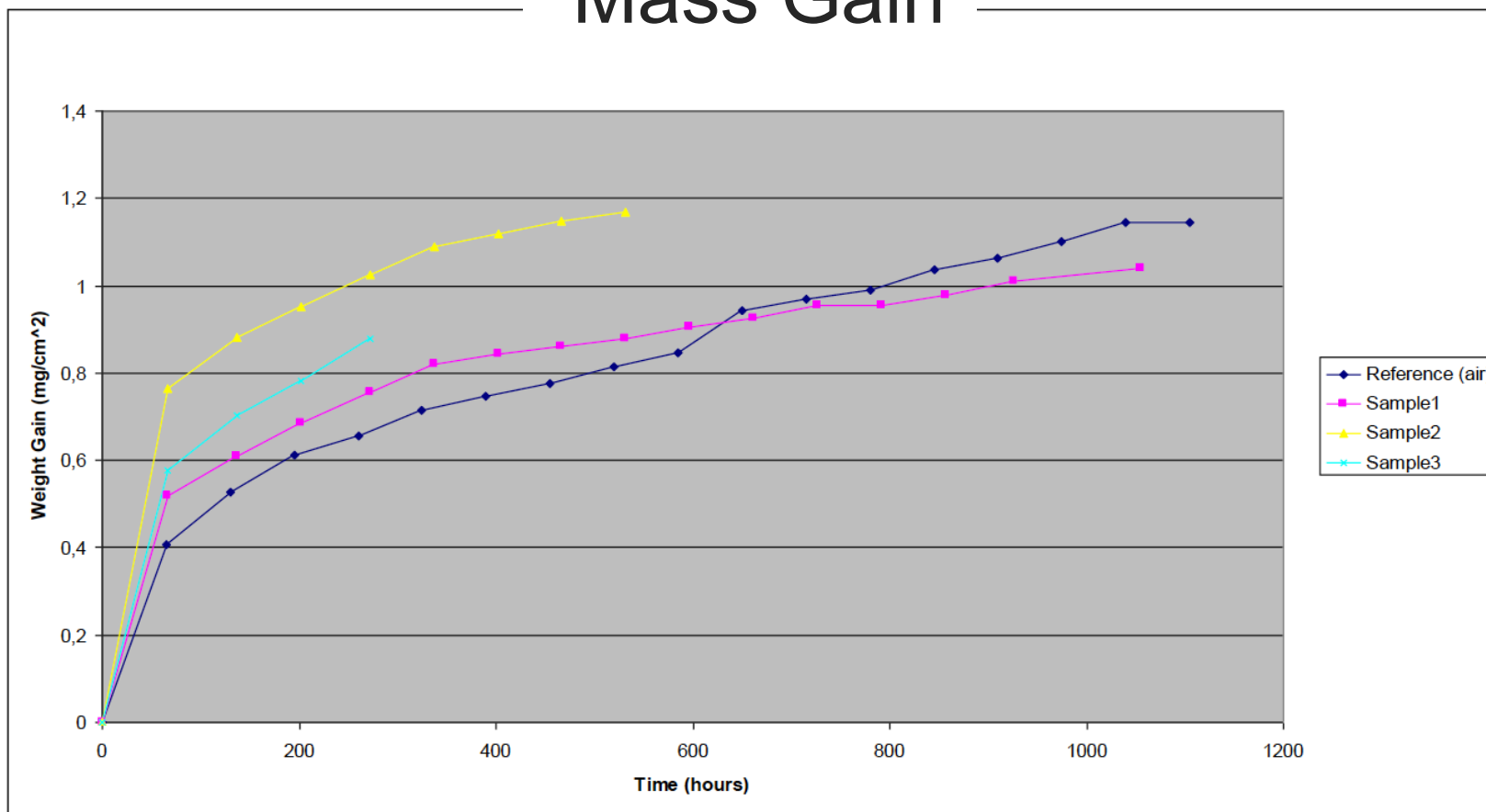
Methods

- Exposure environment:
90% CO₂ - 10% H₂O 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.
- Materials analysis:
SEM, EDX, FIB/SEM,
TEM/STEM



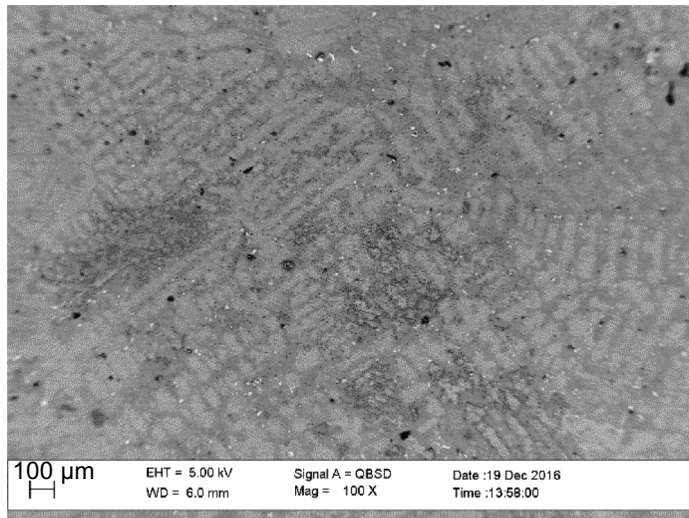
CM247

Mass Gain

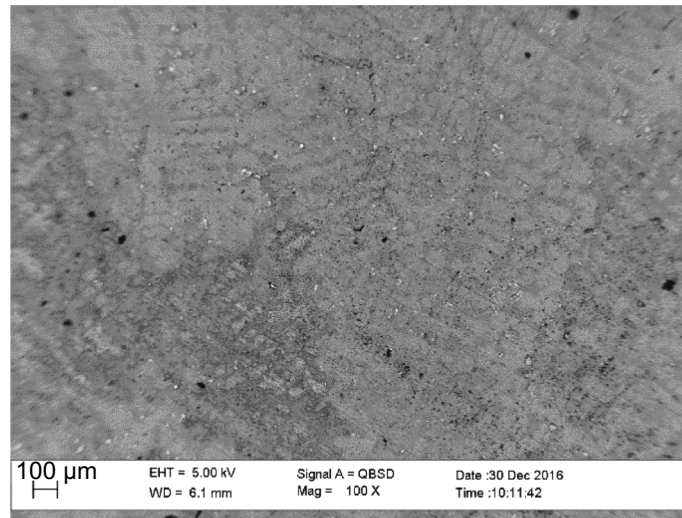


Surface morphology

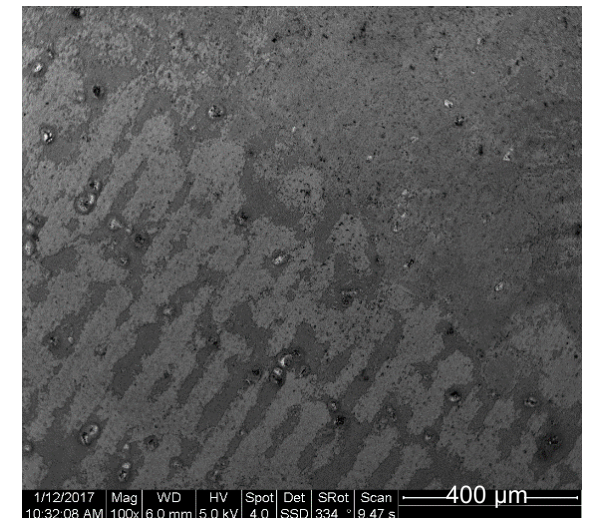
270 h



532 h



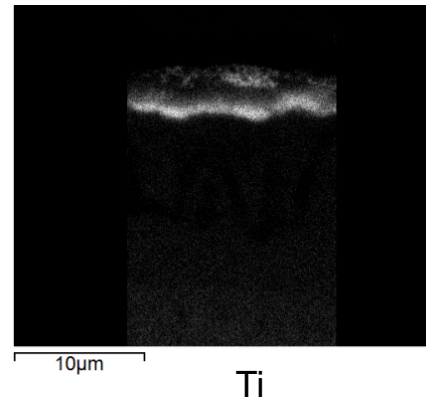
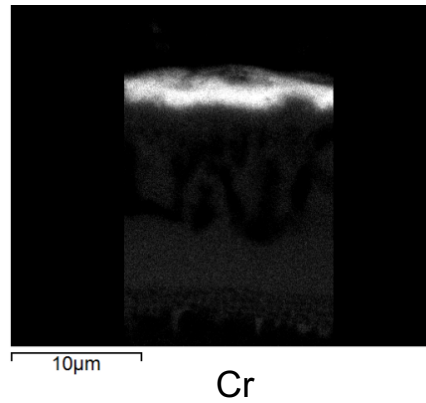
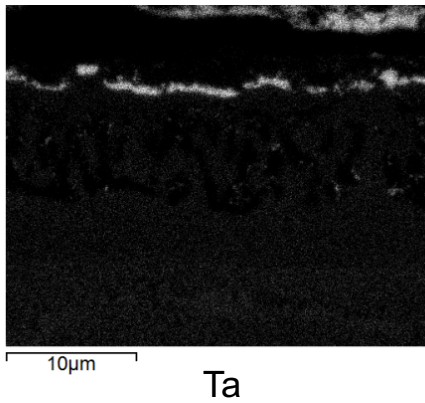
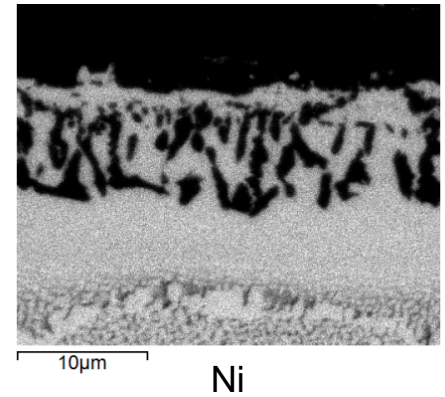
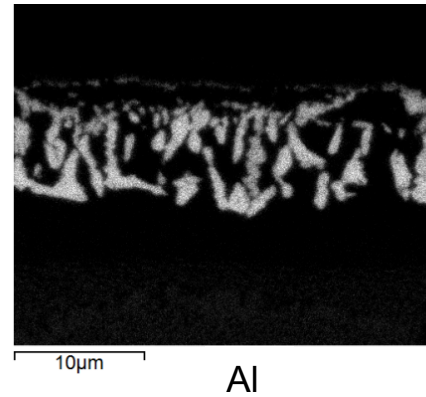
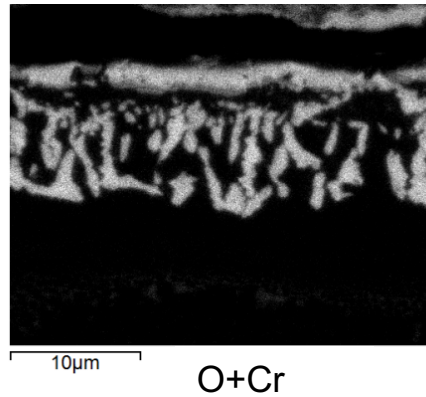
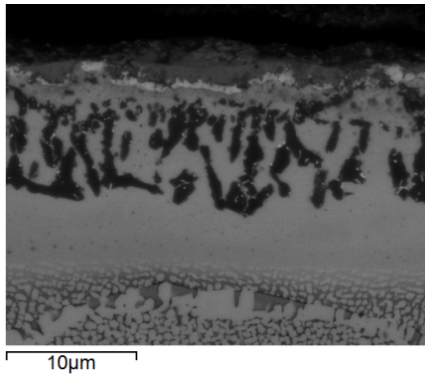
1056 h



CM247

90% CO₂ - 10% H₂O 900°C

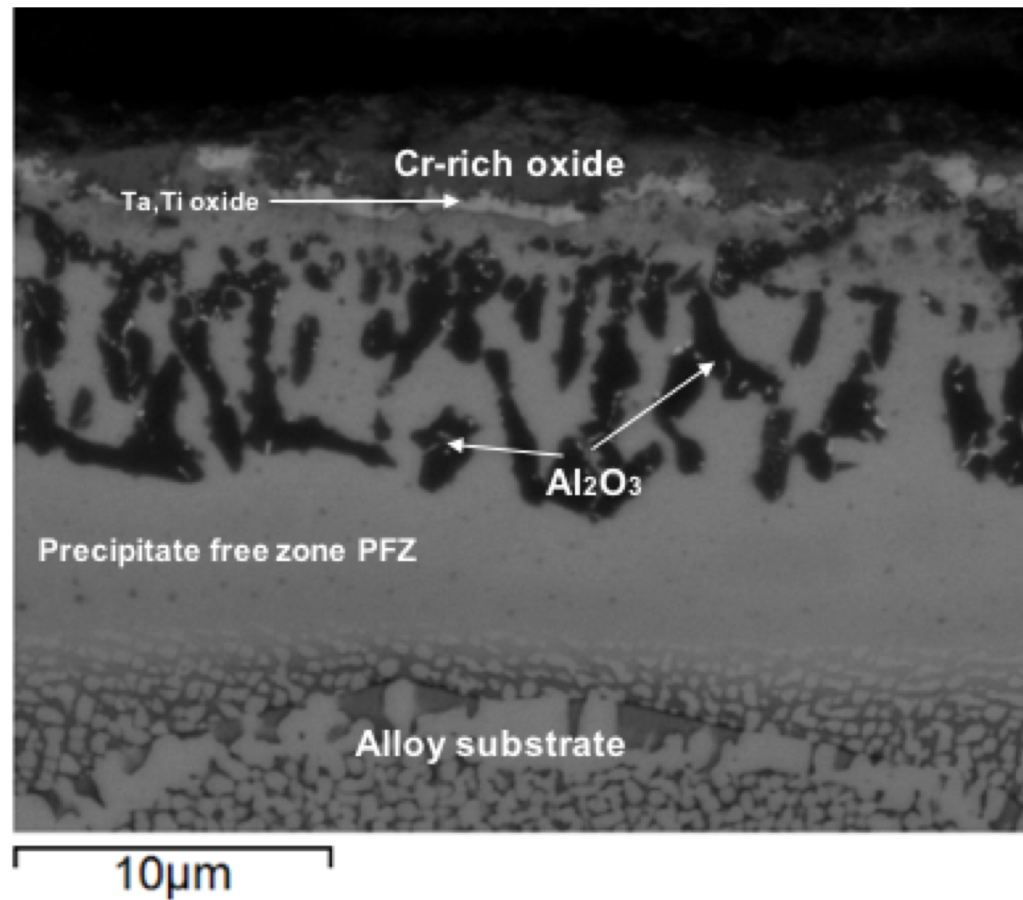
Thick oxide region



CM247 270 h

90% CO₂ - 10% H₂O 900°C

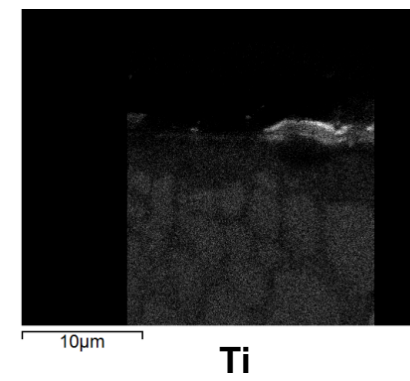
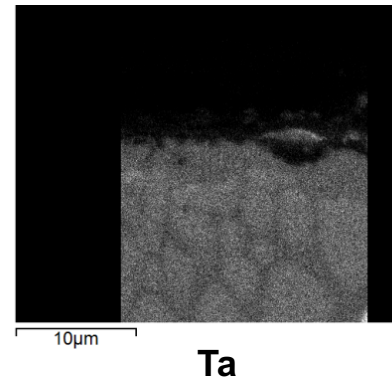
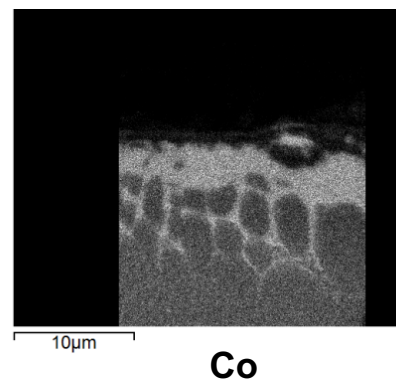
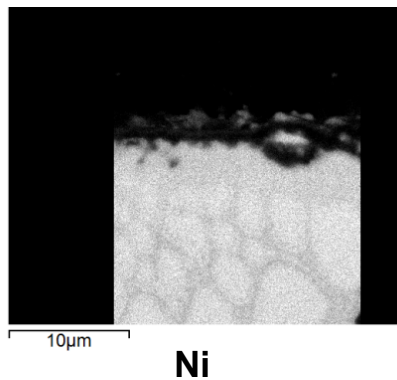
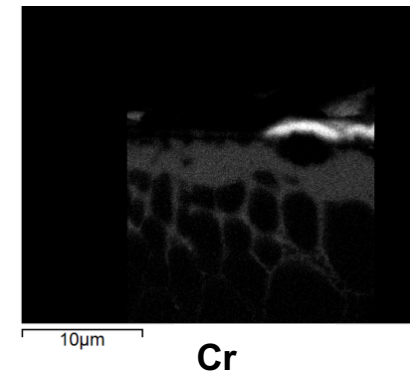
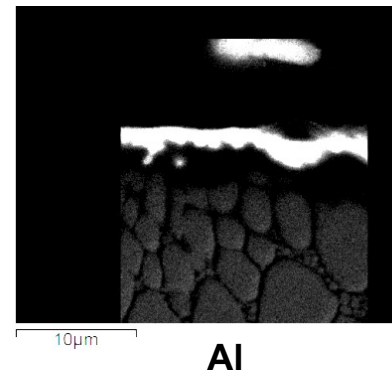
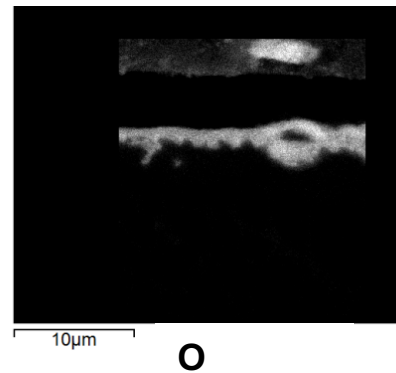
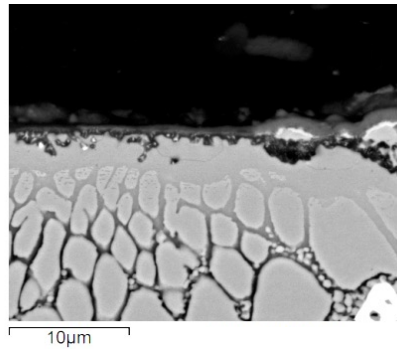
Thick oxide region



CM247 270 h

90% CO_2 - 10% H_2O 900°C

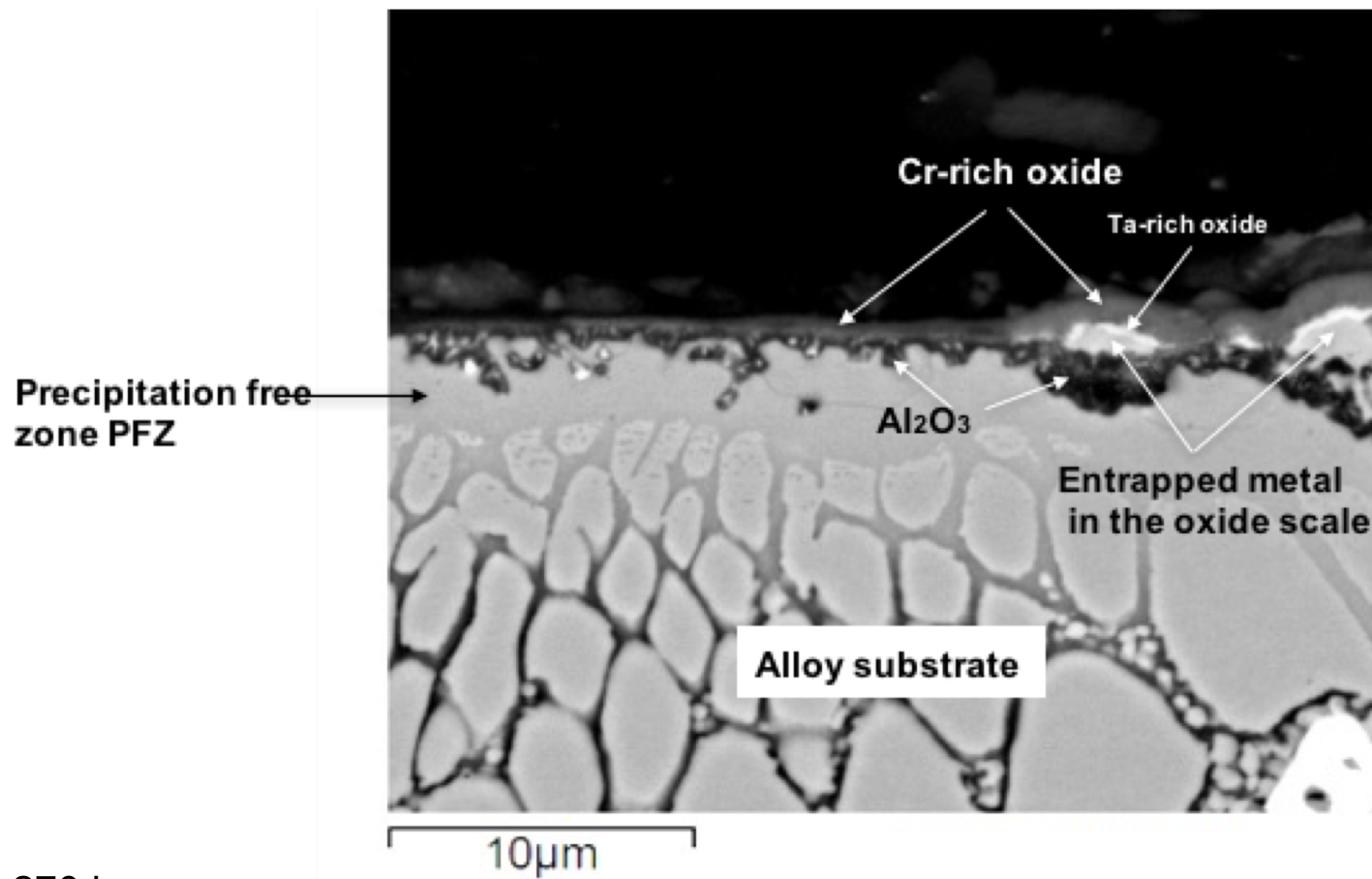
Thin oxide region



CM247 270 h

90% CO₂ - 10% H₂O 900°C

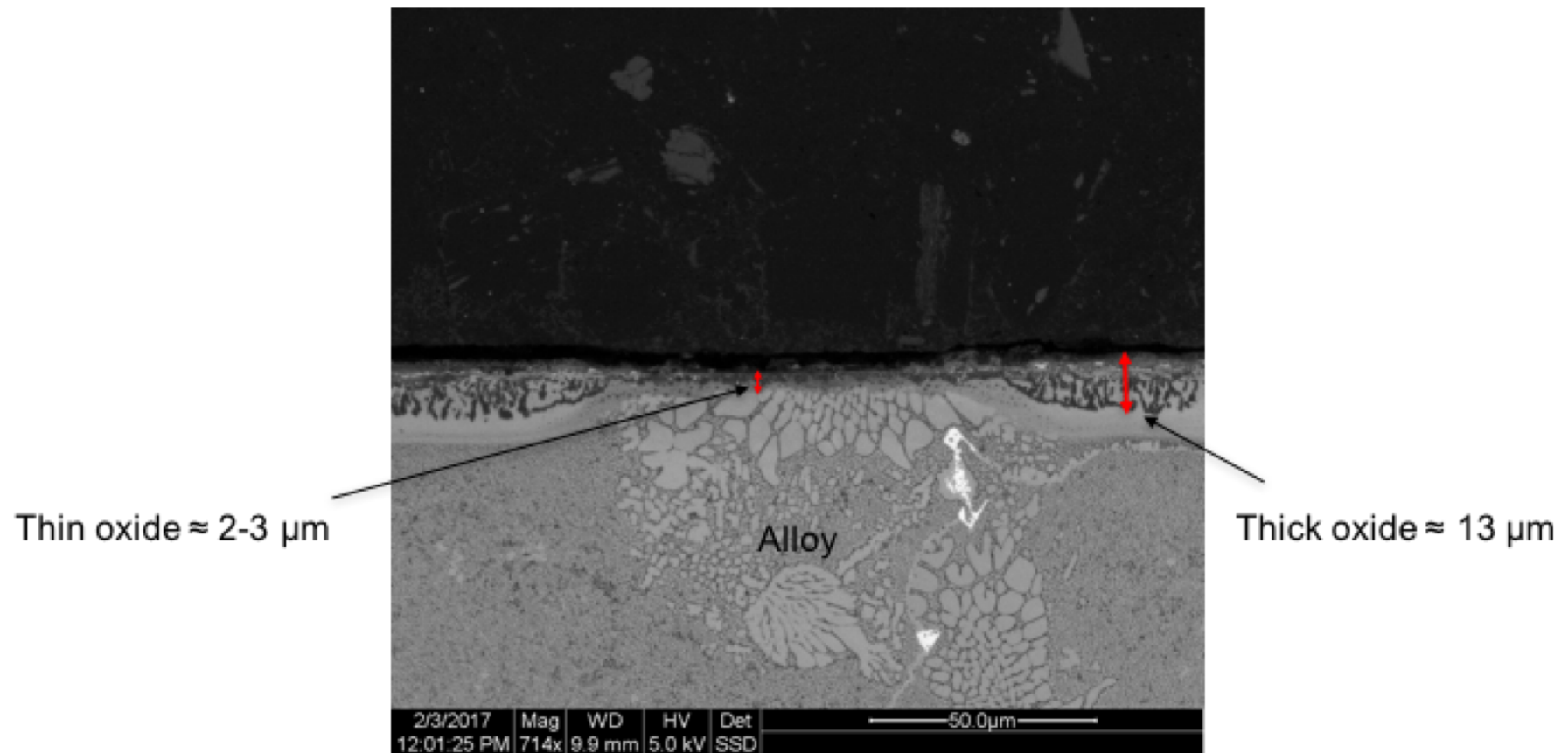
Thin oxide region



CM247 270 h

90% CO_2 - 10% H_2O 900°C

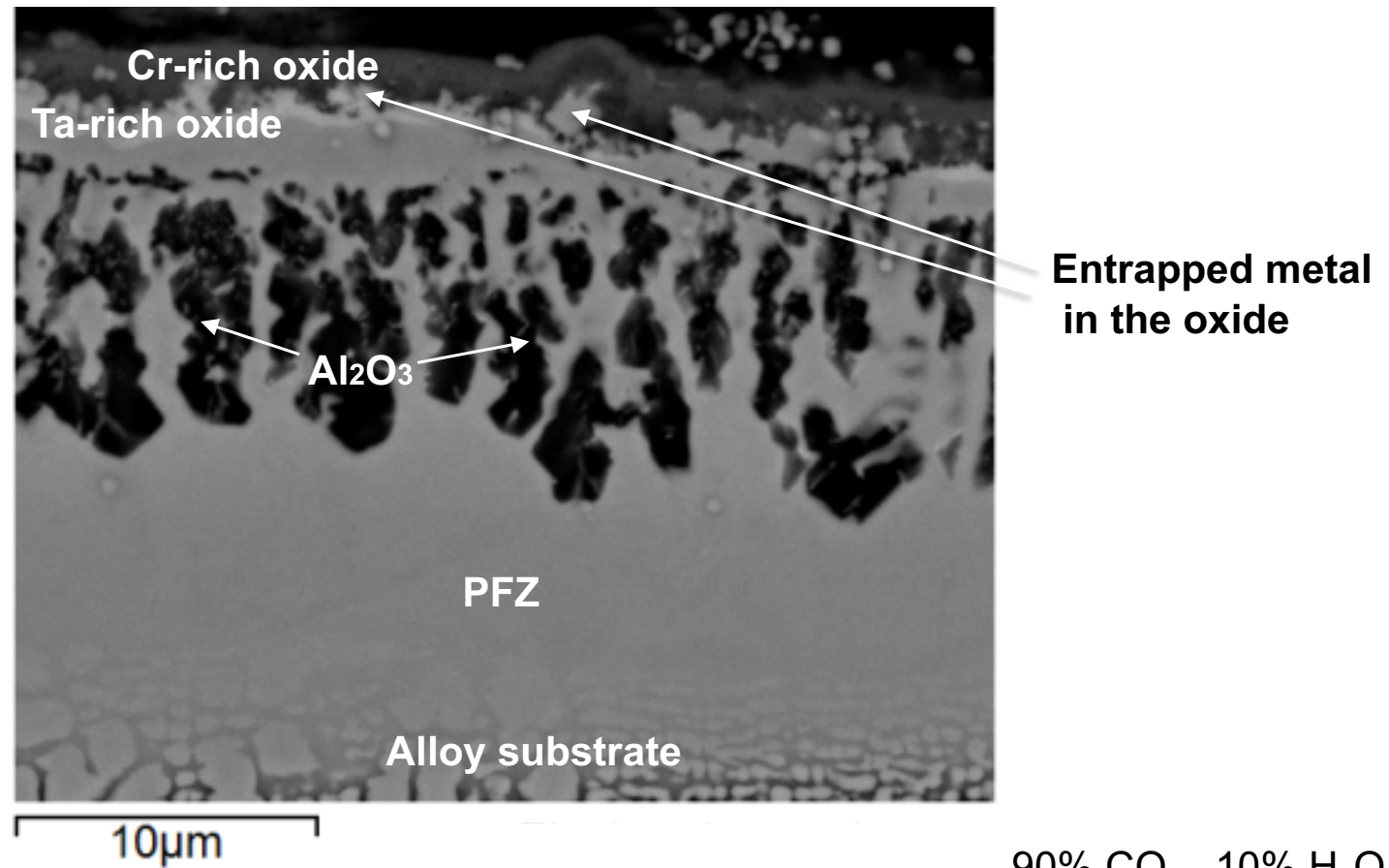
Local alloy inhomogeneity



CM247 270 h

90% CO_2 - 10% H_2O 900°C

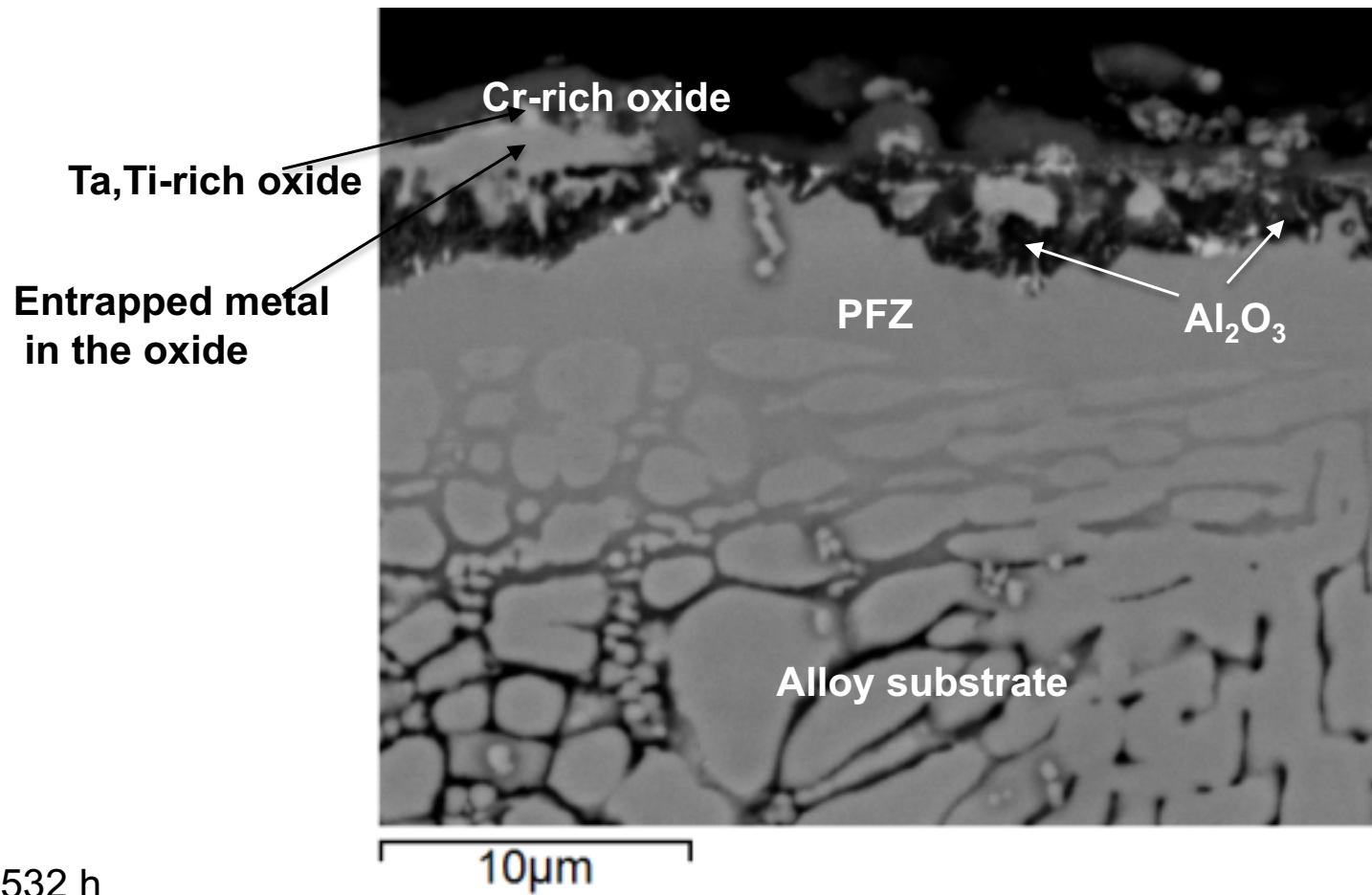
Thick oxide



CM247 532 h

90% CO₂ - 10% H₂O 900°C

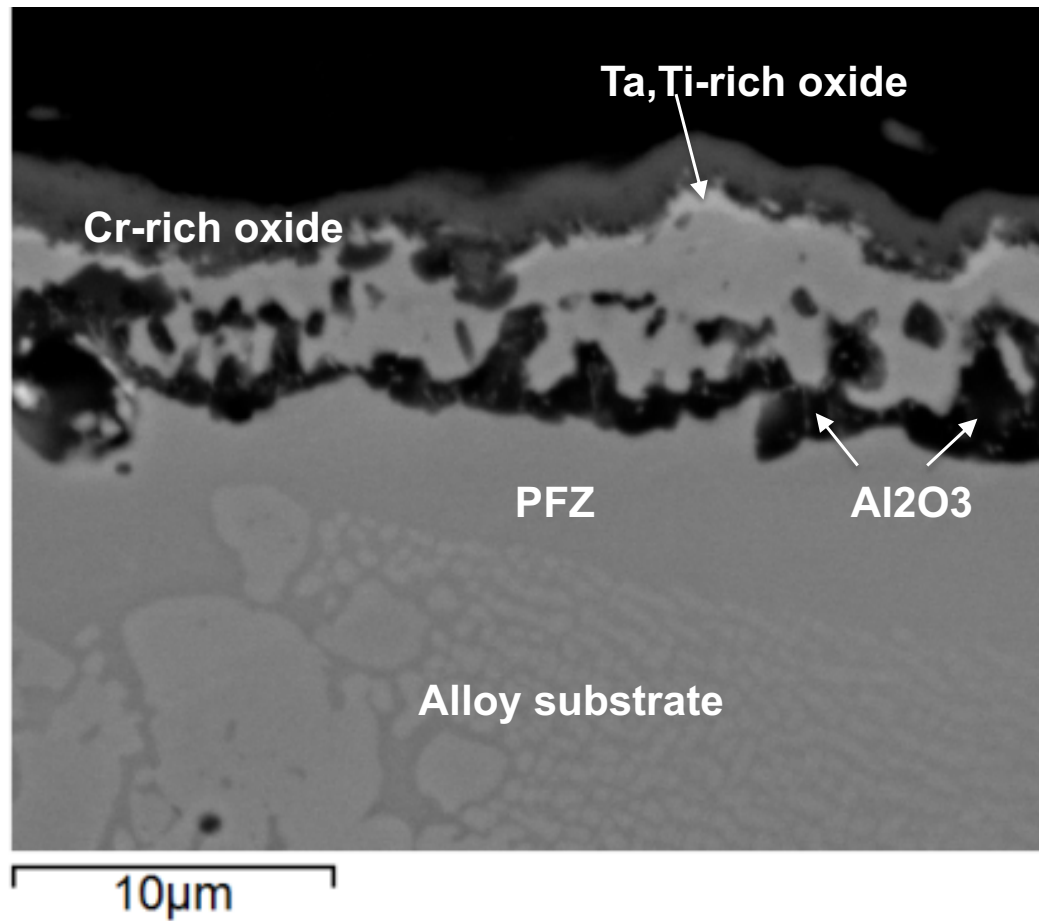
Thin oxide



CM247 532 h

90% CO₂ - 10% H₂O 900°C

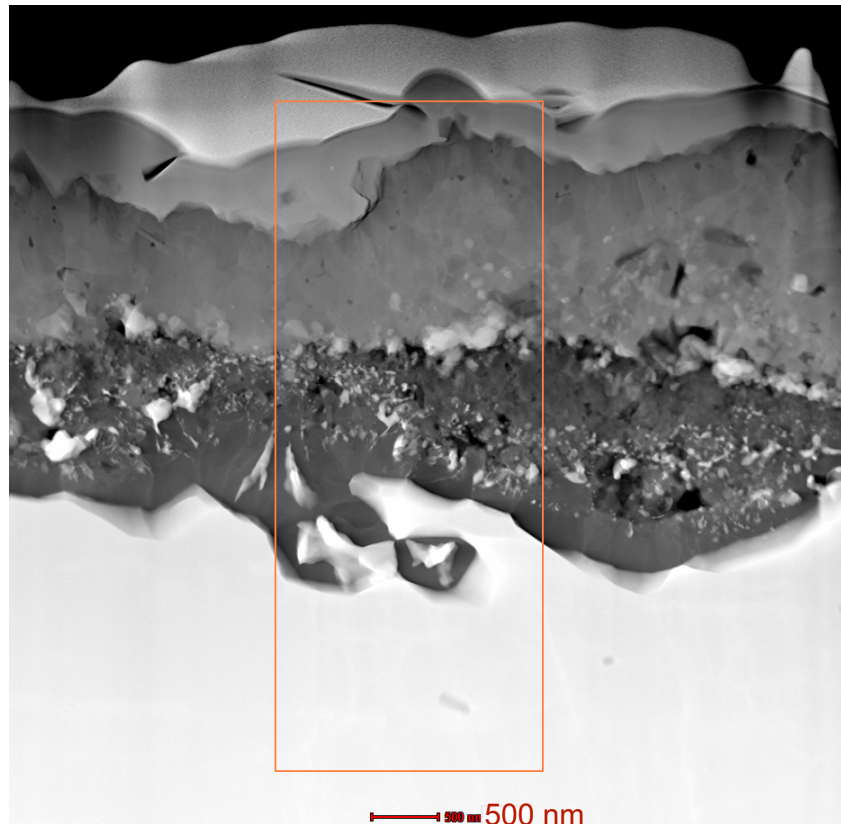
Thick oxide



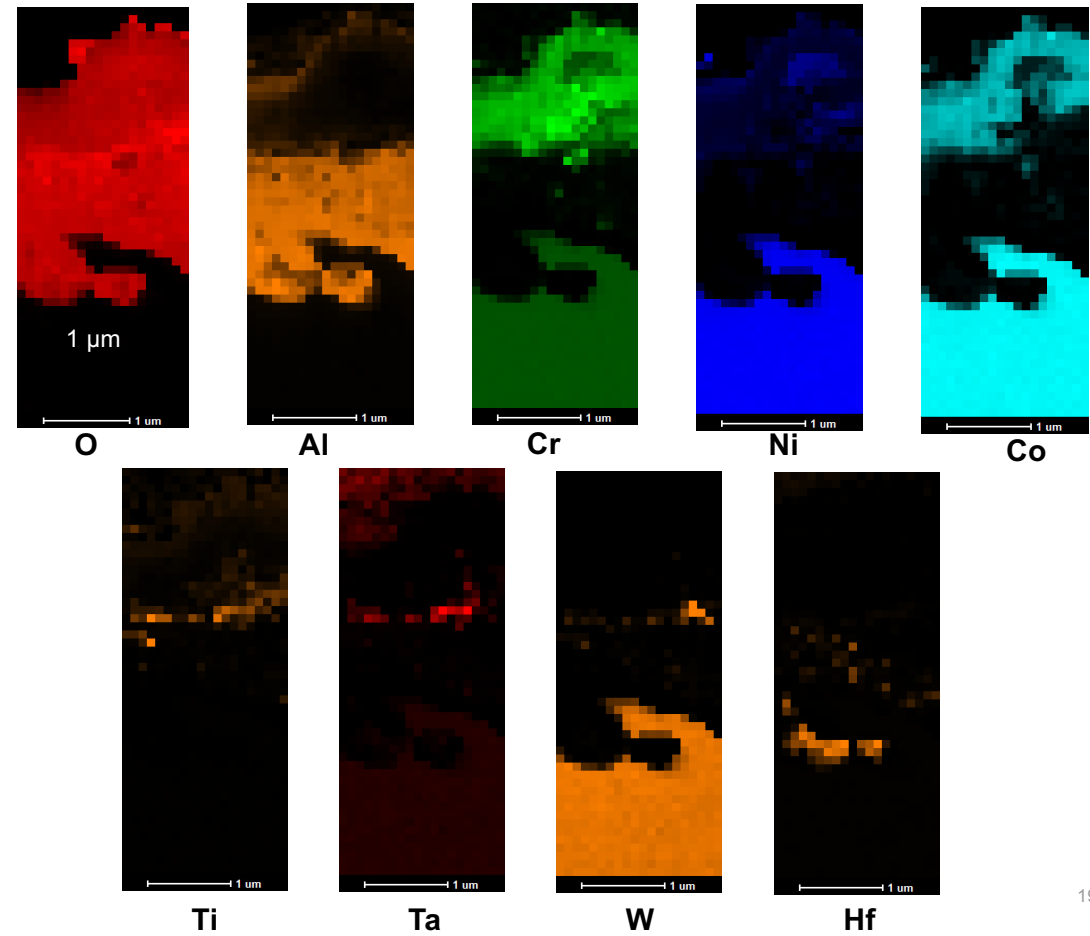
CM247 1056 h

90% CO₂ - 10% H₂O 900°C

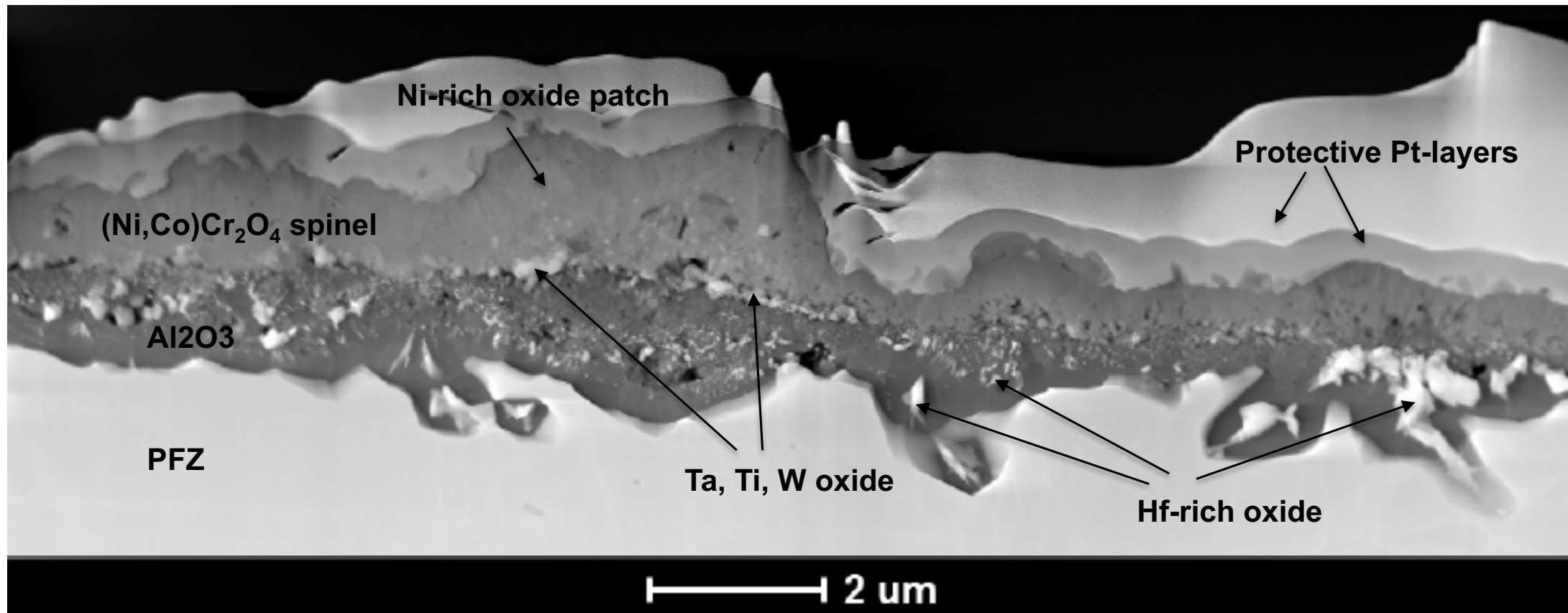
STEM HAADF / EDX maps



CM247 1056 h



CM247 overview



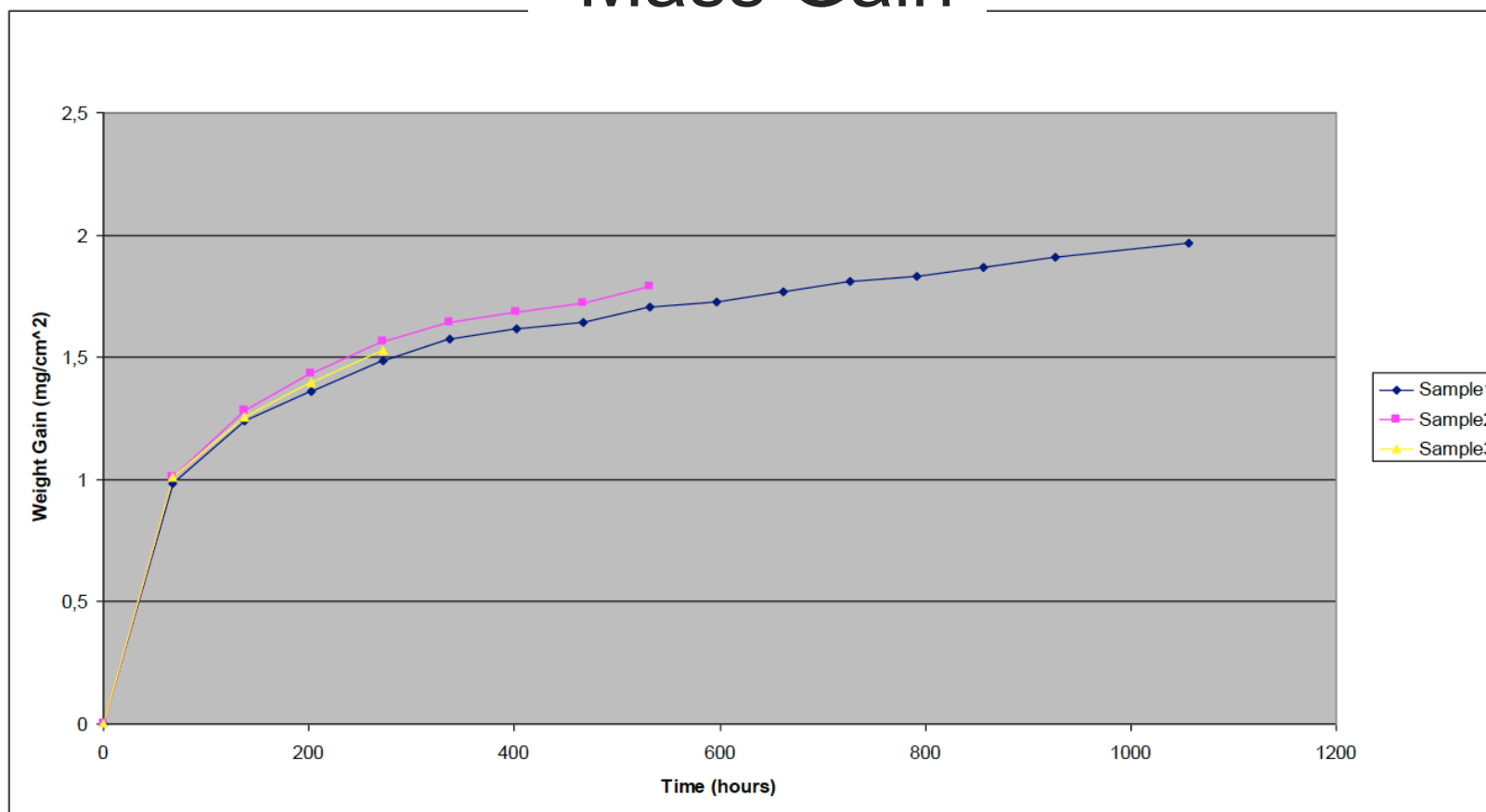
CM247 1056 h

STEM HAADF

90% CO₂ - 10% H₂O 900°C

STAL 15 single crystal

Mass Gain

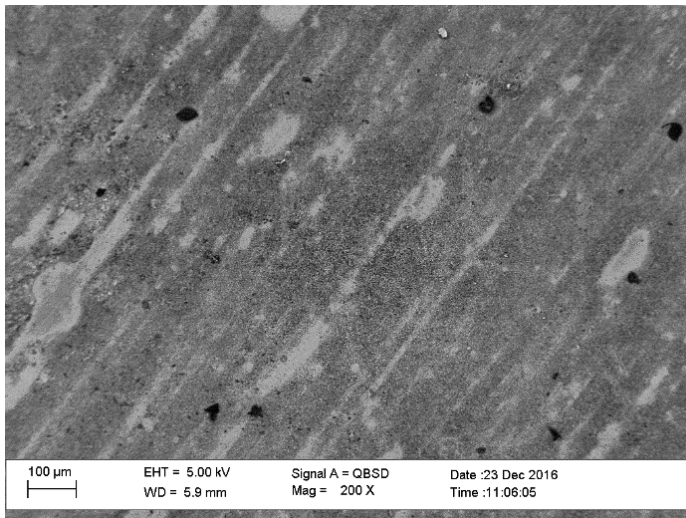


STAL 15

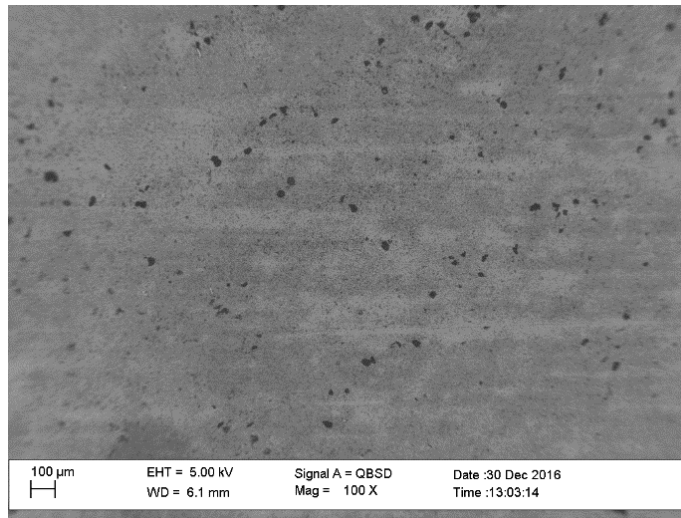
90% CO₂ - 10% H₂O 900°C

Surface morphology

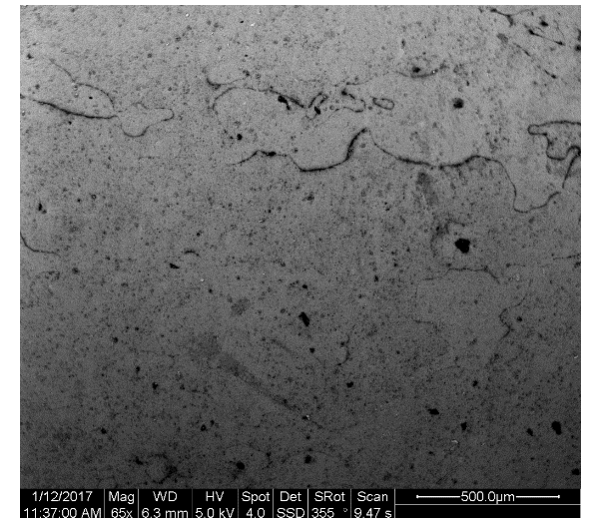
270h



532h



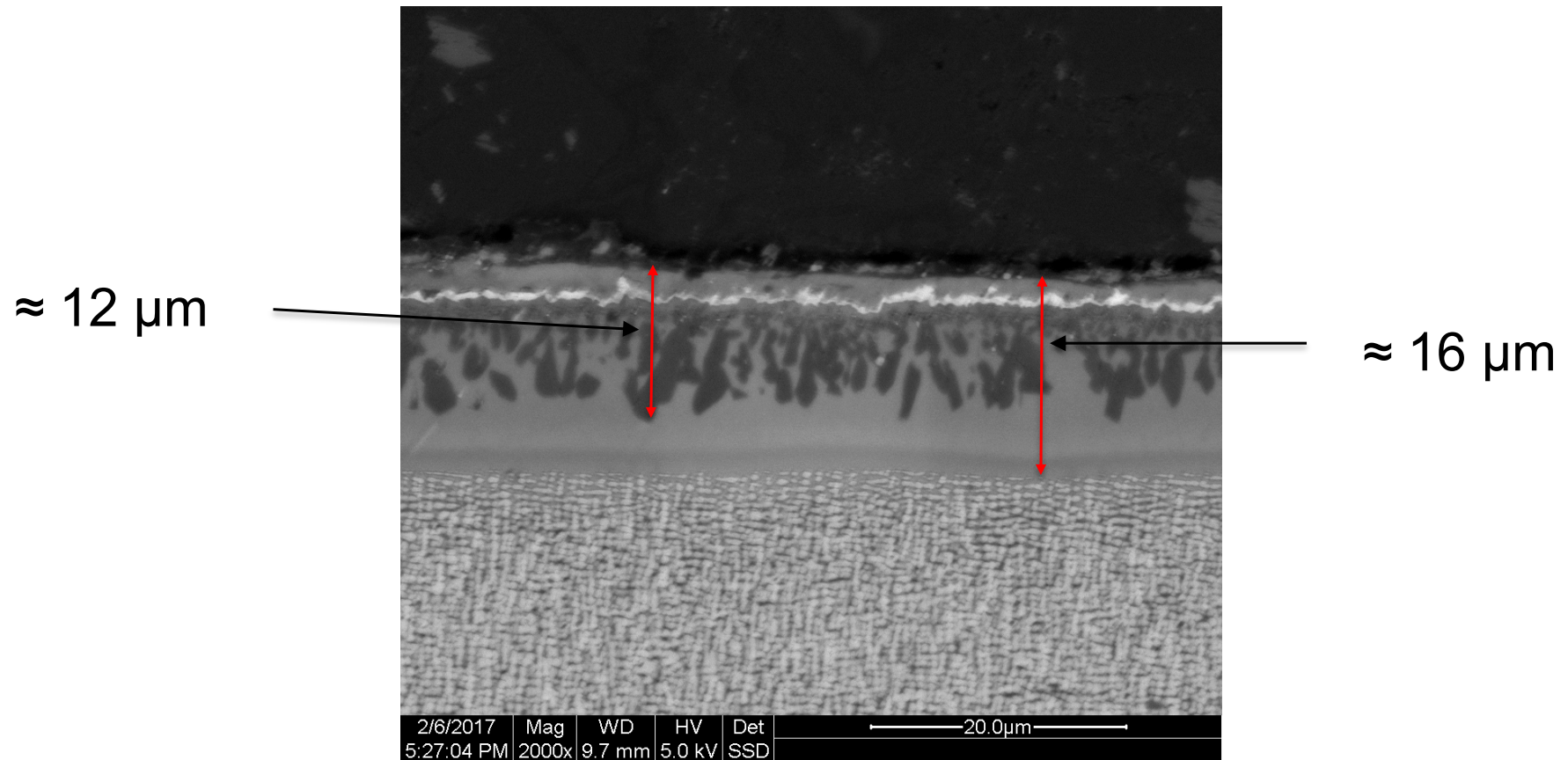
1056h



STAL 15

90% CO₂ - 10% H₂O 900°C

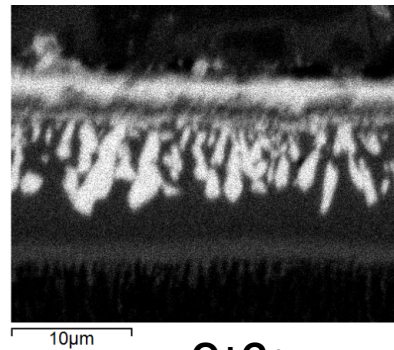
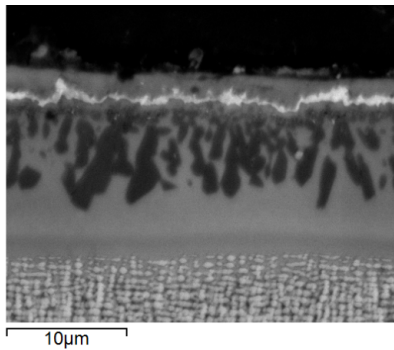
Oxide scale cross-section



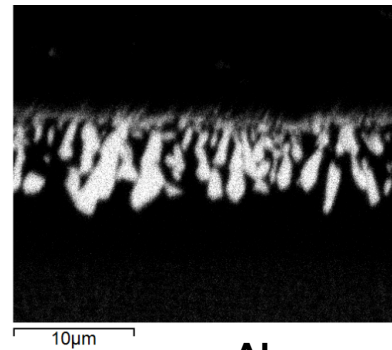
STAL 15 270 h

90% CO_2 - 10% H_2O 900°C

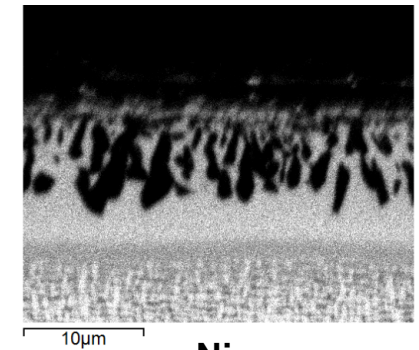
Cross-section SEM/EDX maps



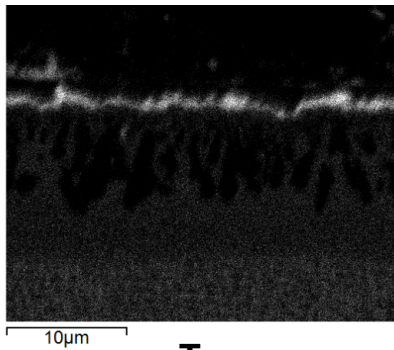
O+Cr



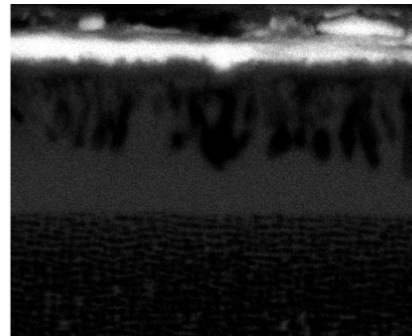
Al



Ni



Ta

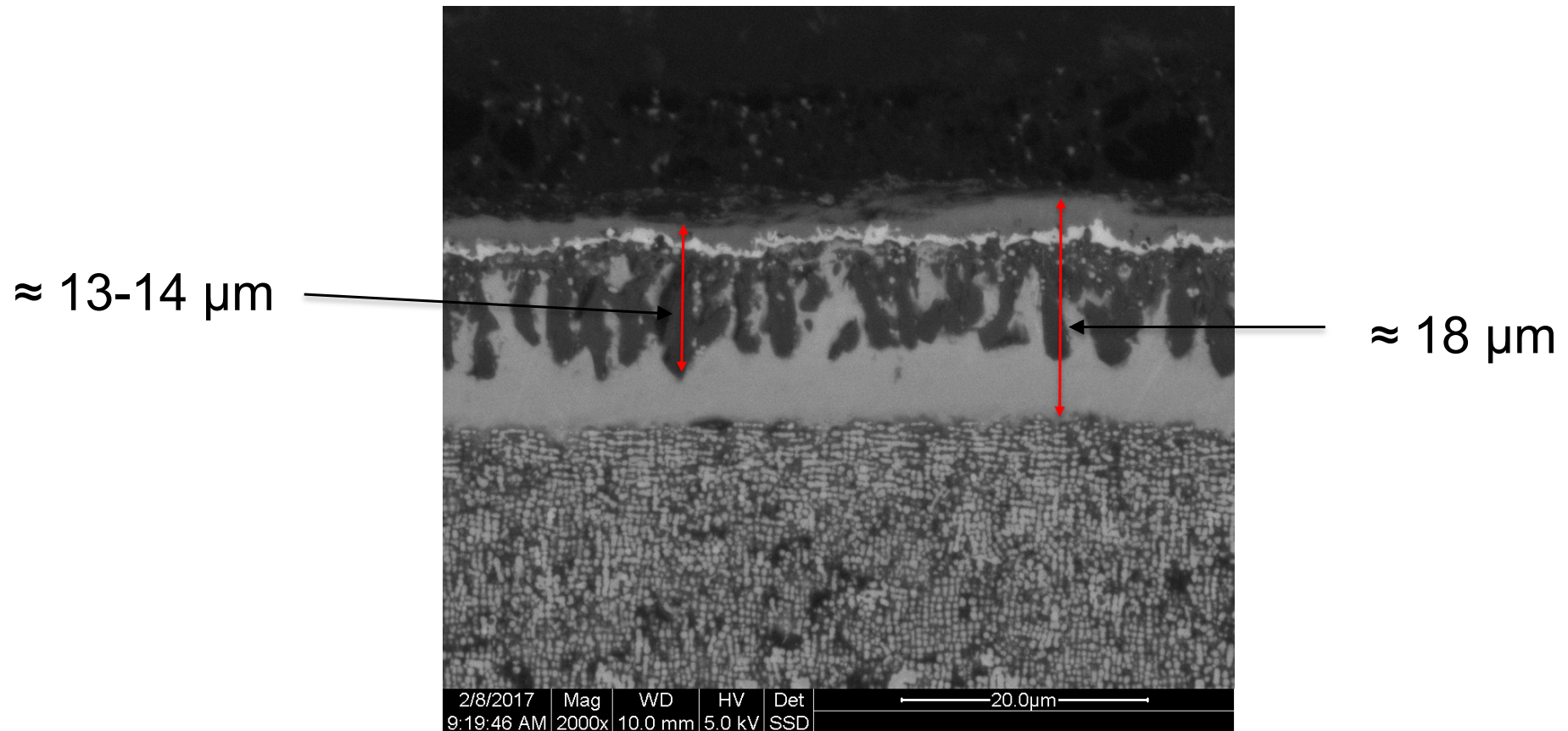


Cr

STAL 15 270 h

90% CO₂ - 10% H₂O 900°C

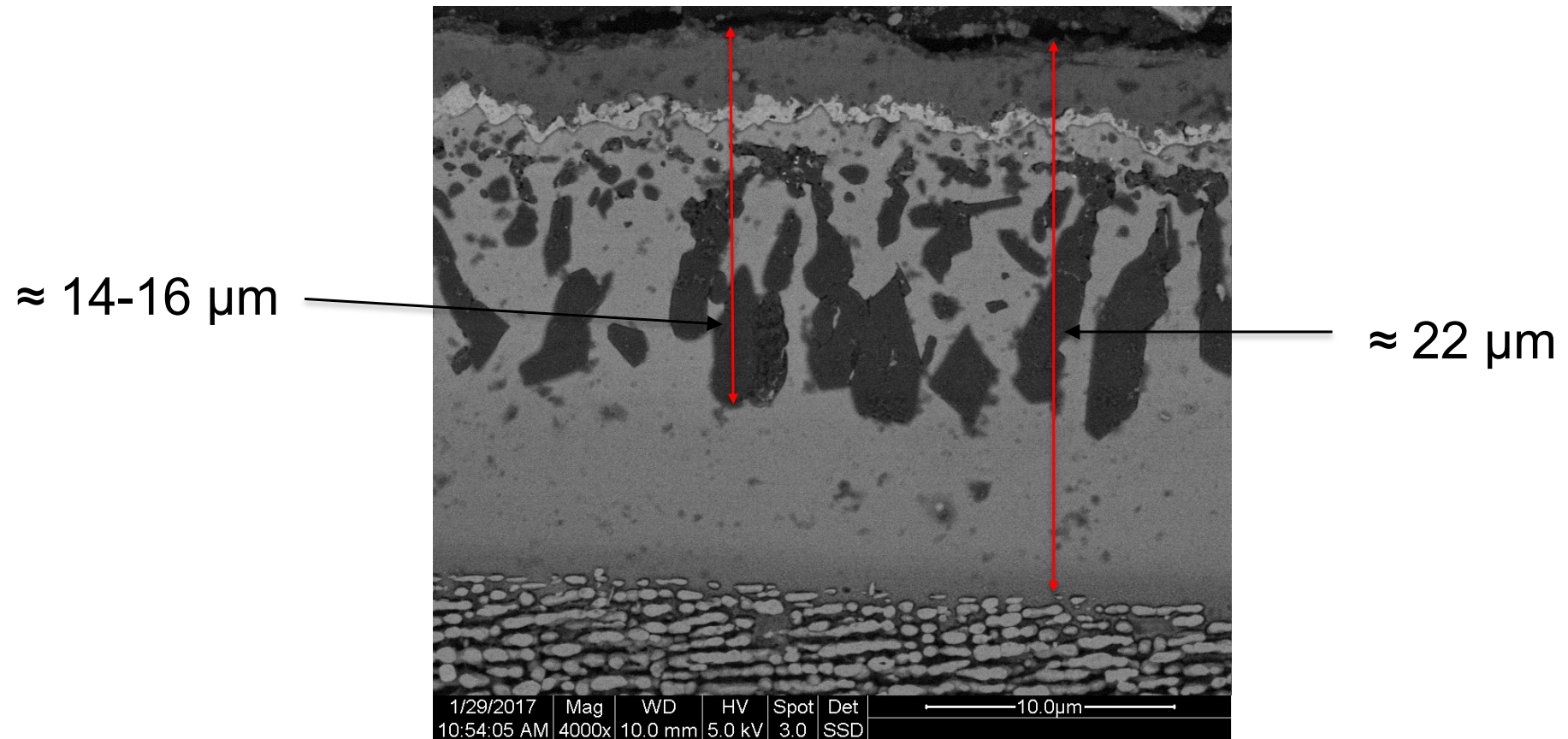
Oxide scale cross-section



STAL 15 532 h

90% CO₂ - 10% H₂O 900°C

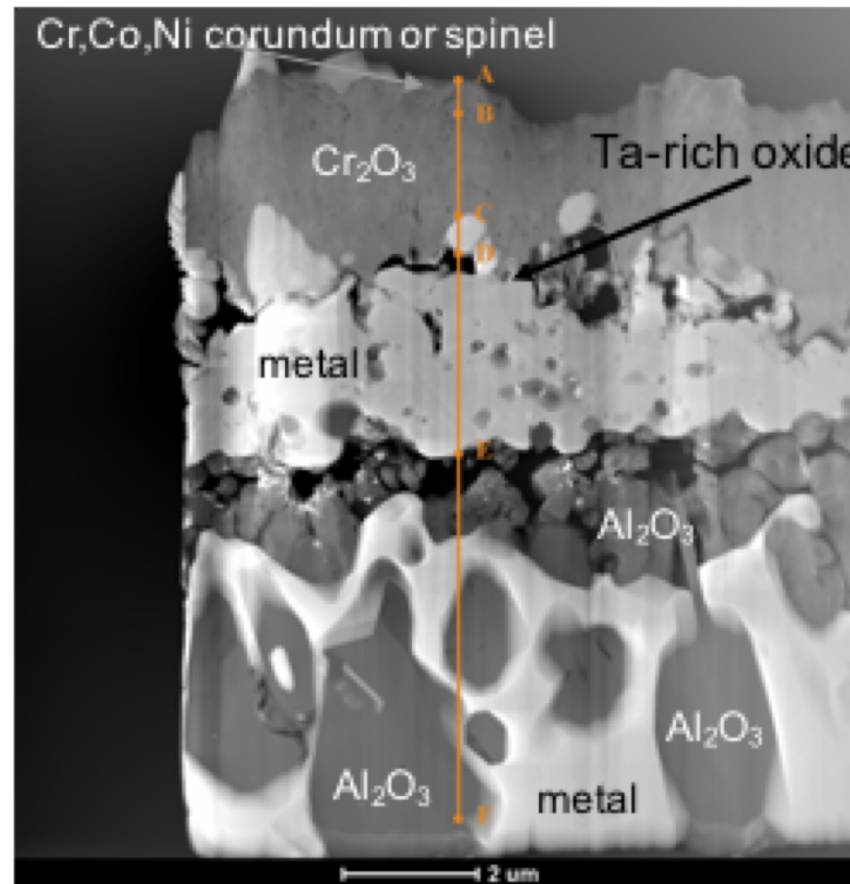
Oxide scale cross-section



STAL 15 1056 h

90% CO₂ - 10% H₂O 900°C

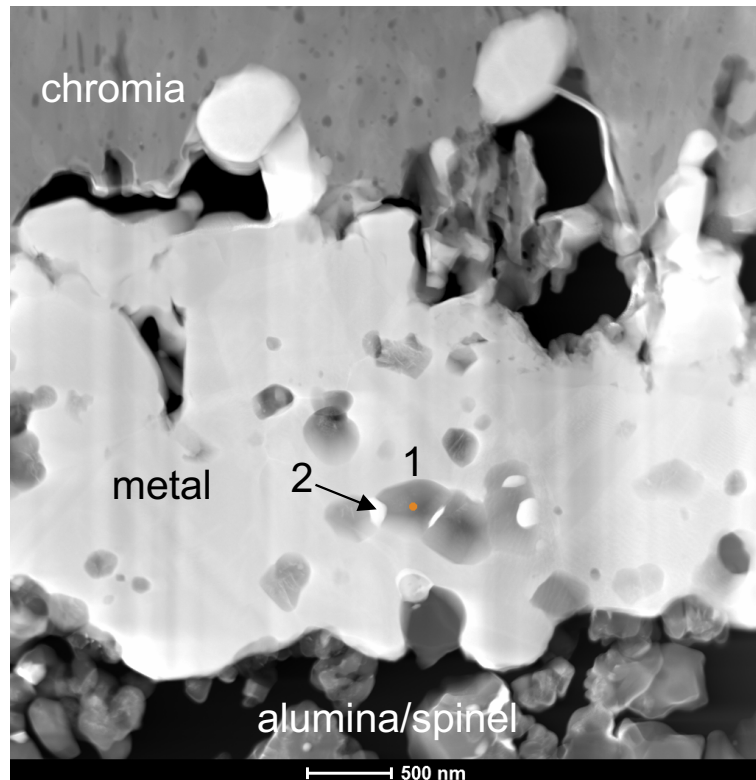
STEM HAADF cross-section



STAL 15 1056 h

90% CO_2 - 10% H_2O 900°C

STEM HAADF /EDX data



1) Ni-Co-Al-Cr-Ta spinel

Point 1

Element	Weight %	Atomic %
-----	-----	-----
Al(K)	31.68	54.60
Cr(K)	8.77	7.84
Co(K)	3.83	3.02
Ni(K)	37.46	29.67
Mo(K)	0.86	0.42
Ta(L)	11.54	2.96
W(L)	5.82	1.47

2) Hf-rich oxide
(white)

Point 2

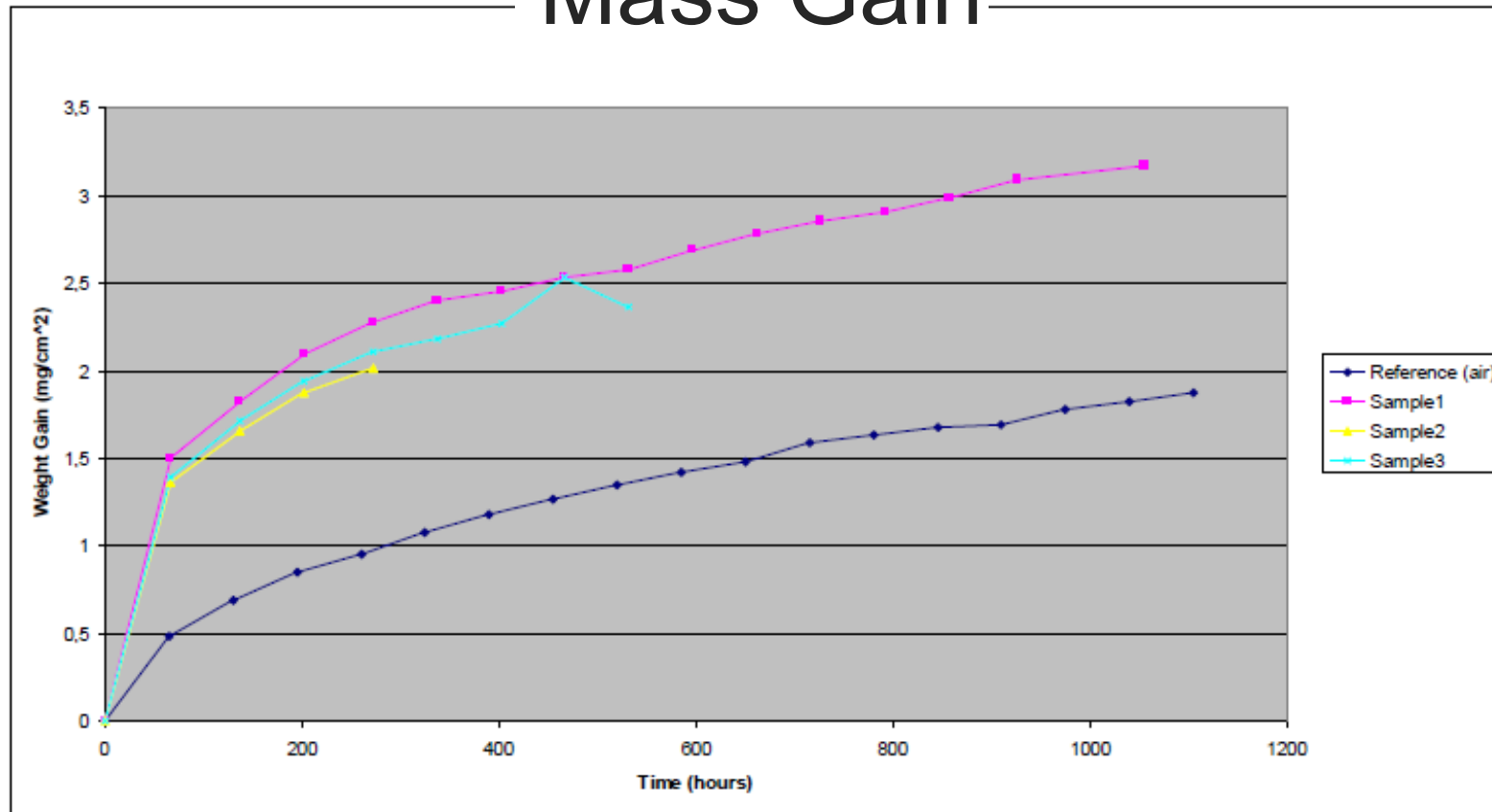
Element	Weight %	Atomic %
-----	-----	-----
Cr(K)	6.30	11.15
Co(K)	4.10	6.39
Ni(K)	34.17	53.51
Mo(K)	1.10	1.05
Hf(L)	41.09	21.16
Ta(L)	13.22	6.71

STAL 15 1056 h

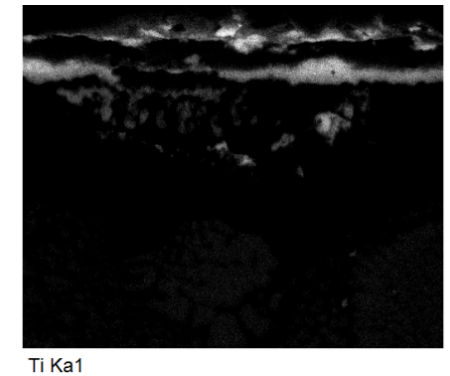
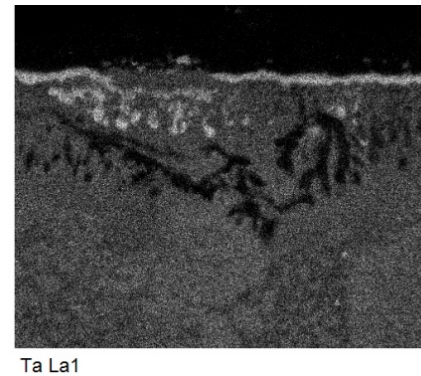
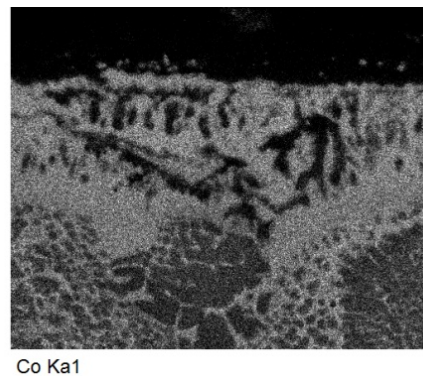
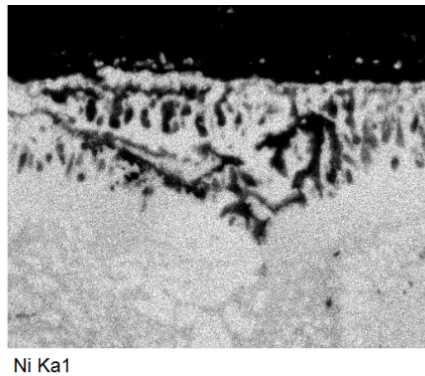
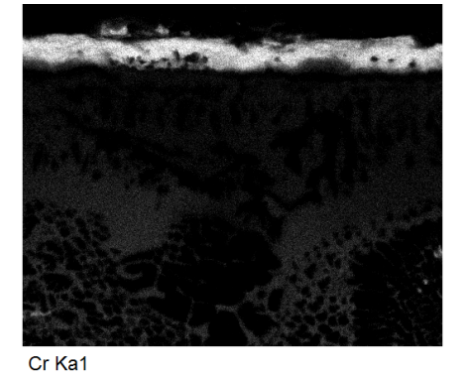
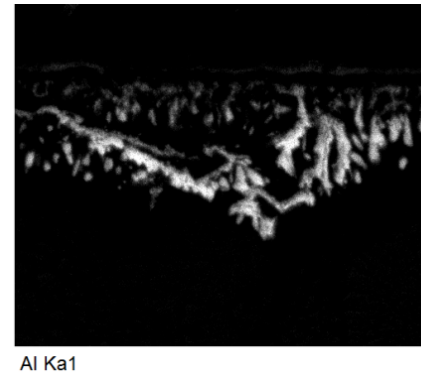
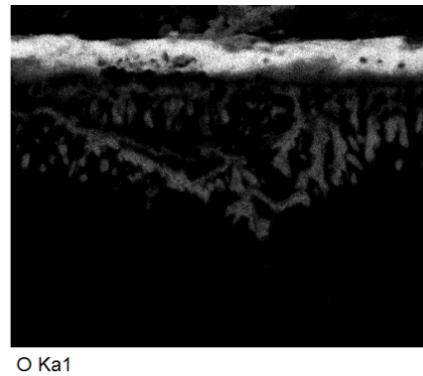
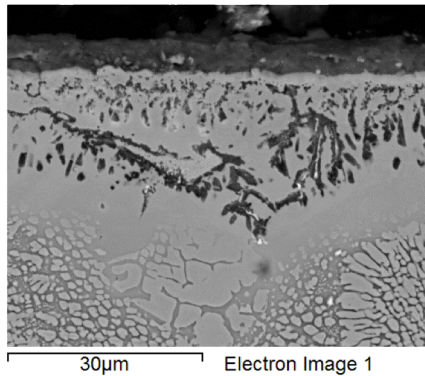
90% CO₂ - 10% H₂O 900°C

IN792

Mass Gain



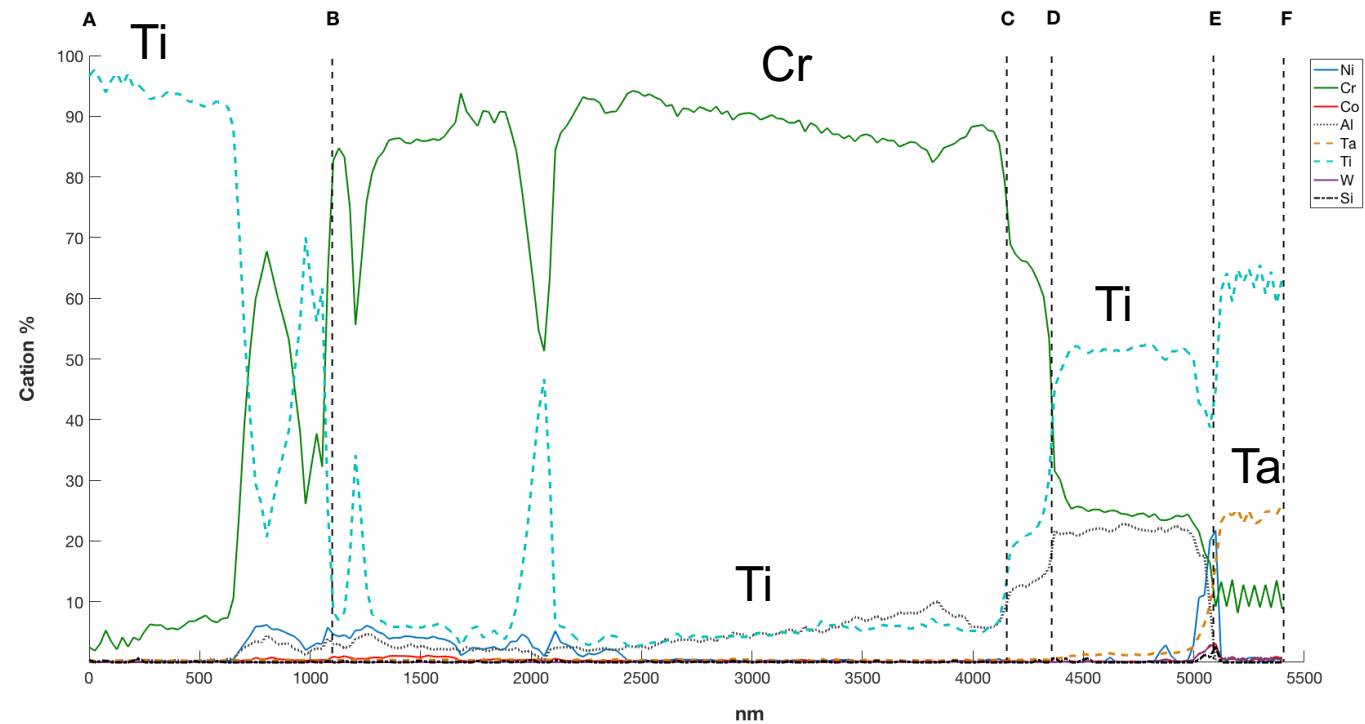
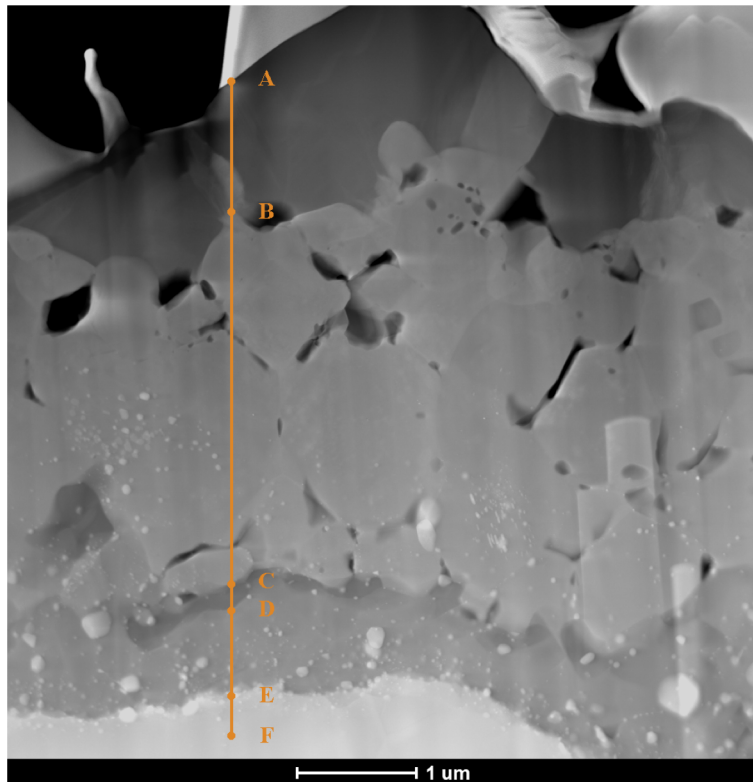
Cross-sectional SEM/EDX elemental maps



IN792 270 h

90% CO₂ - 10% H₂O ³²900°C

STEM-HAADF/EDX cross-section

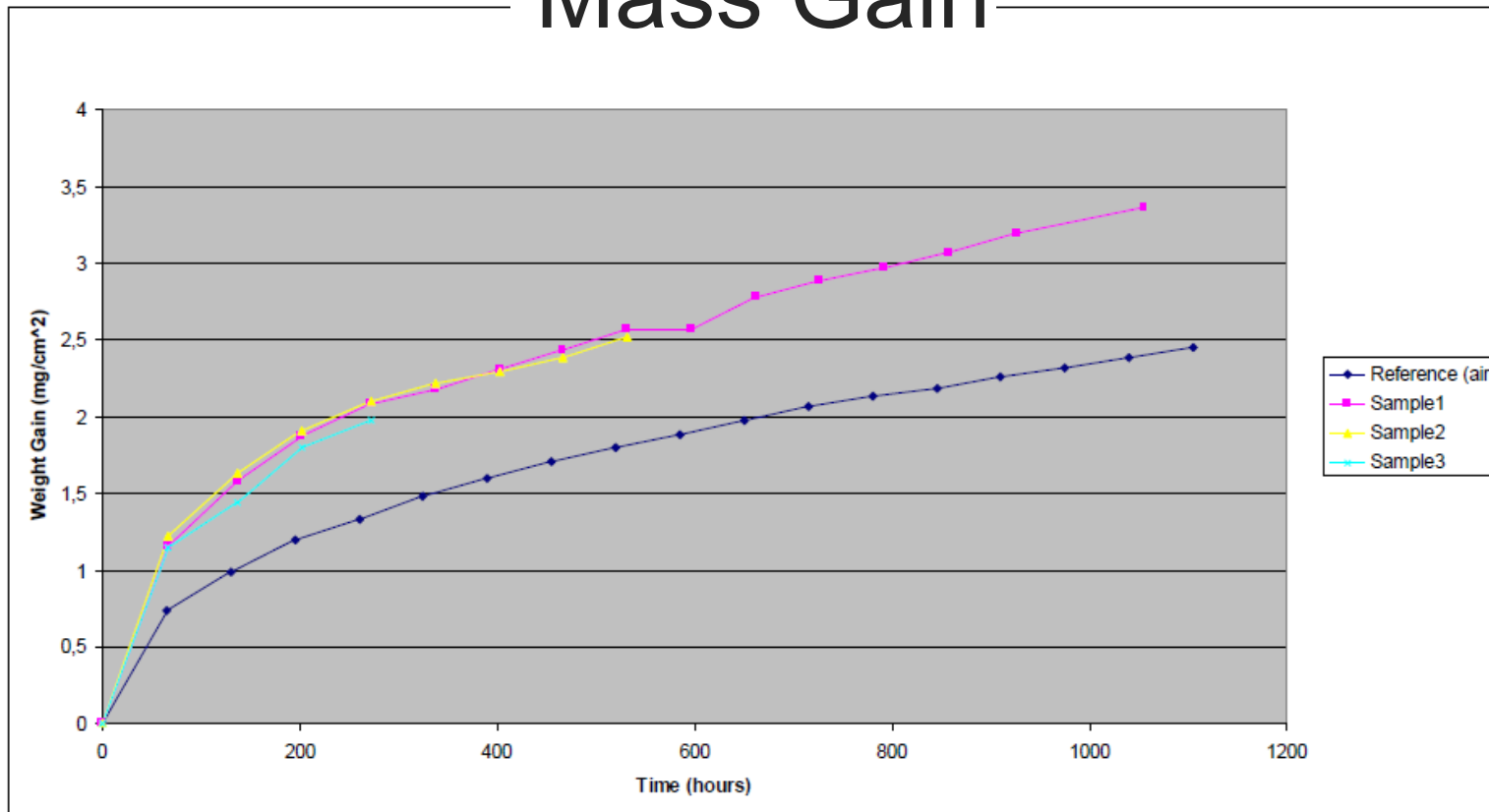


IN792 1056 h

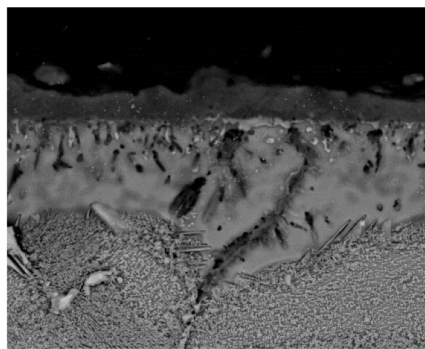
90% CO₂ - 10% H₂O 900°C

IN939

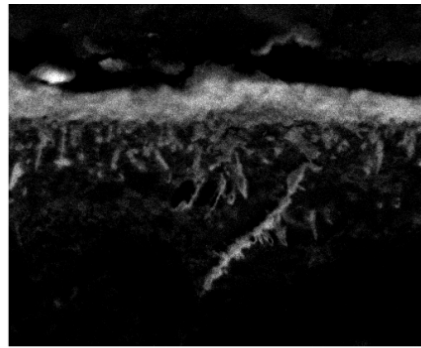
Mass Gain



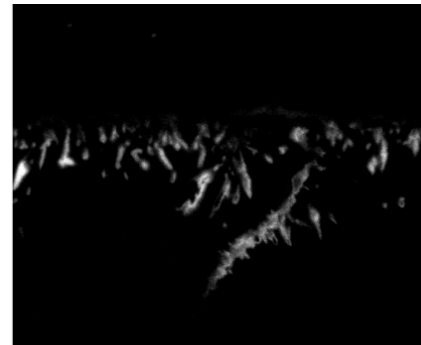
Cross-sectional SEM/EDX elemental maps



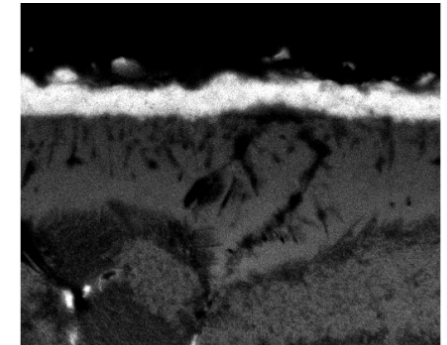
30µm Electron Image 1



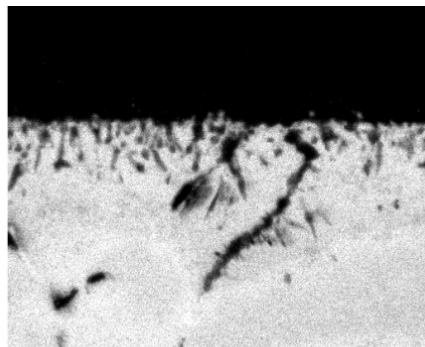
O Ka1



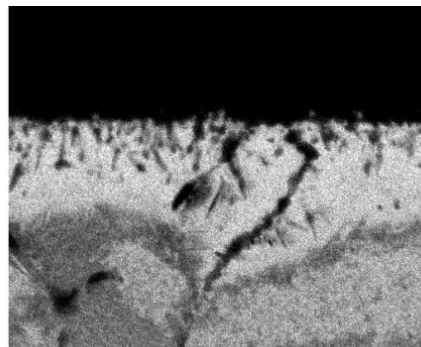
Al Ka1



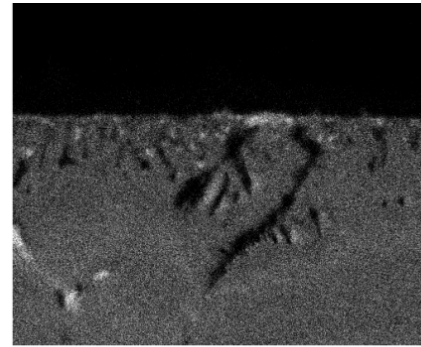
Cr Ka1



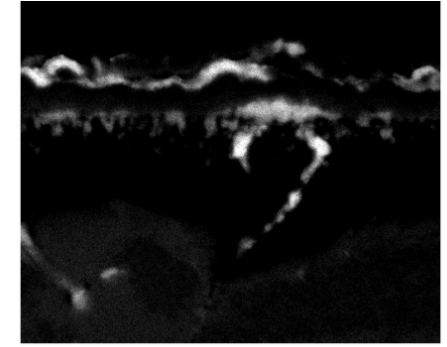
Ni Ka1



Co Ka1



Ta La1

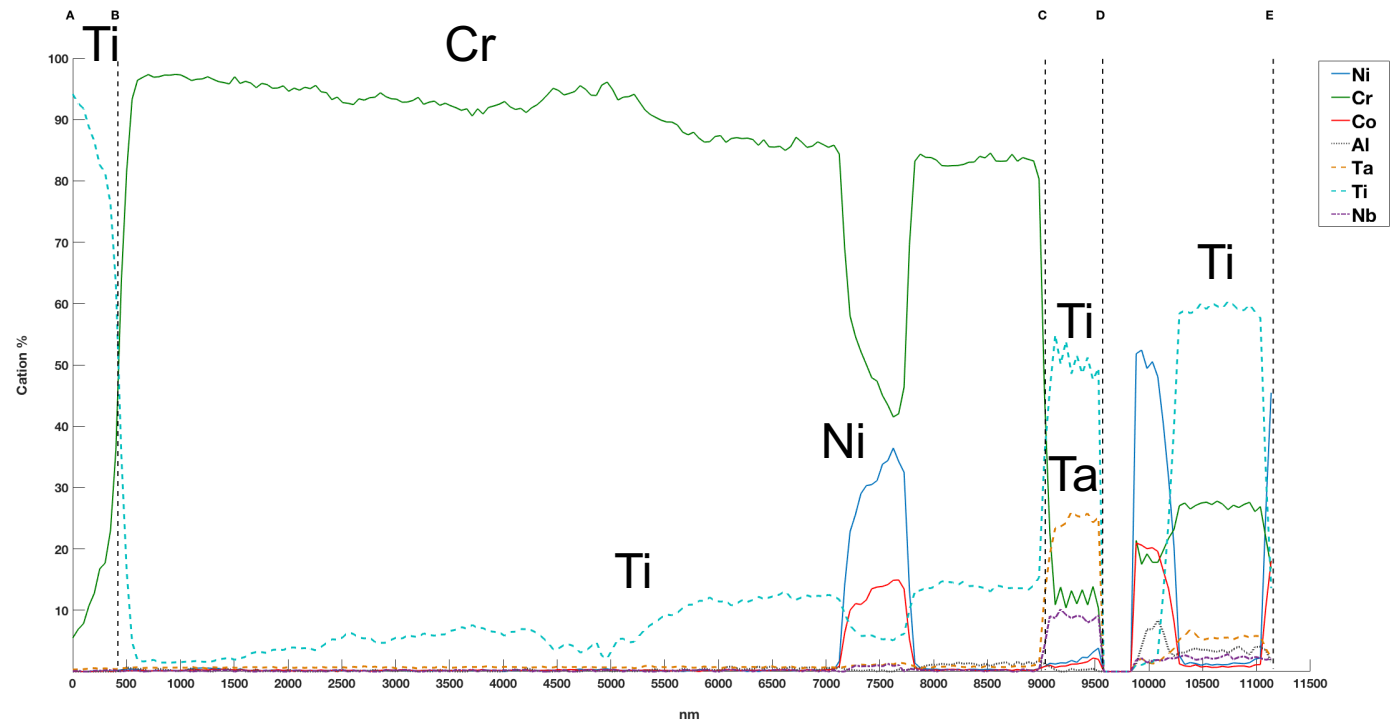
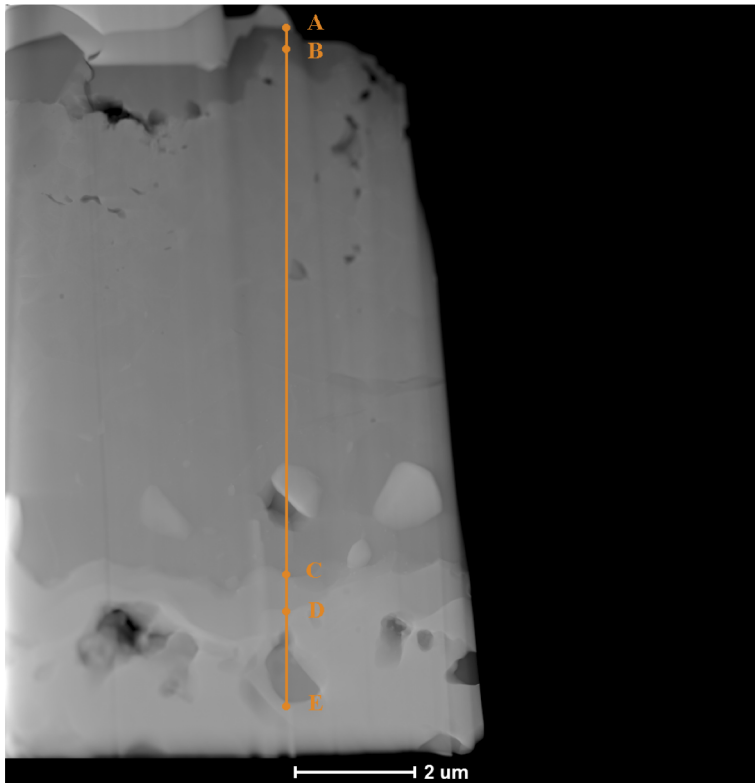


Ti Ka1

IN939 270 h

90% CO₂ - 10% H₂O ³⁶900°C

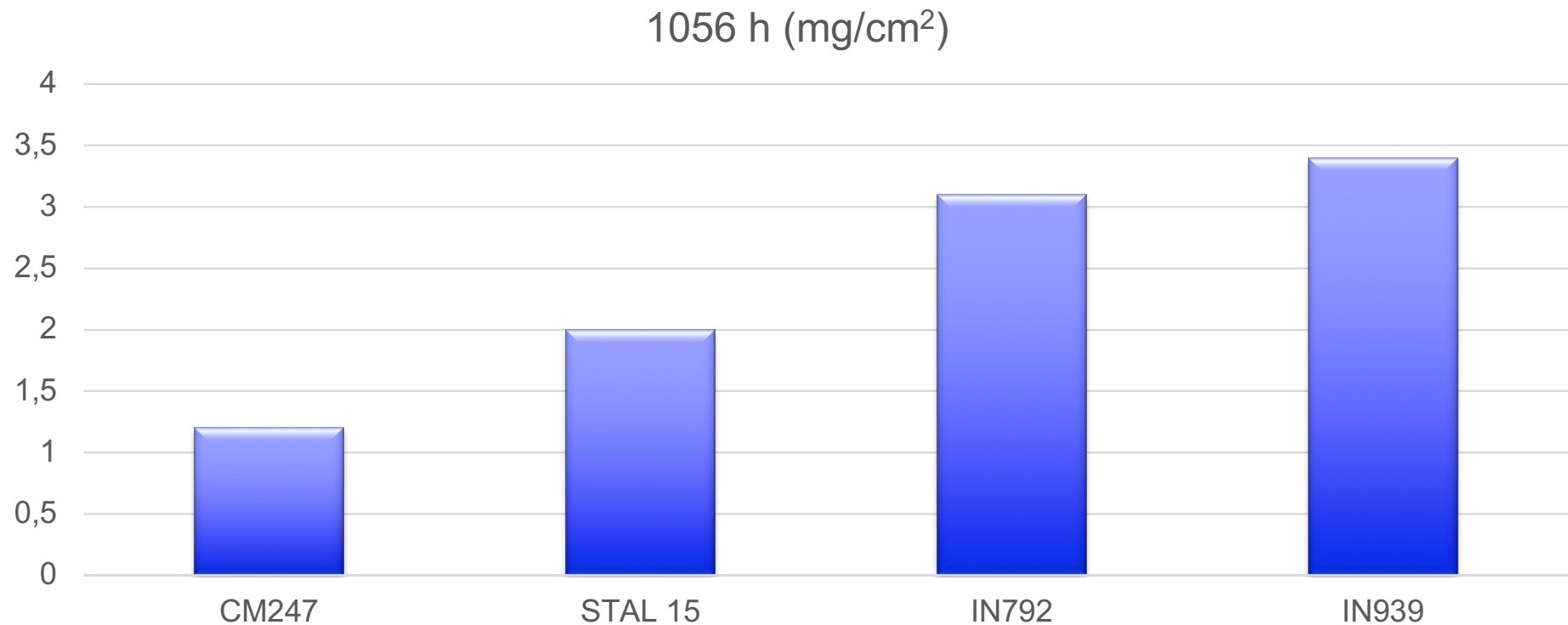
STEM/EDX cross-section



IN939 1056 h

90% CO₂ - 10% H₂O 900°C²⁷

Weight gain comparison



90% CO₂ - 10% H₂O ³⁸ 900°C

Summary and conclusions

- ⊗ Four Ni-base superalloys have been tested in an oxy-fuel-type lab environment; cyclic exposures at 900°C up to 1056 h.
- ⊗ The samples were investigated by a combination of analytical techniques
- ⊗ None of the alloys formed a thin, continuous and protective Al_2O_3 scale
- ⊗ All alloys formed a multilayer scale, including externally grown chromia, spinels and internally grown alumina fingers, with a subjacent precipitation free zone
- ⊗ The results suggest that these alloys need to be coated for applications under the investigated conditions

**Thank you for your
attention**

