Single rod tests at KIT with Cr coated claddings

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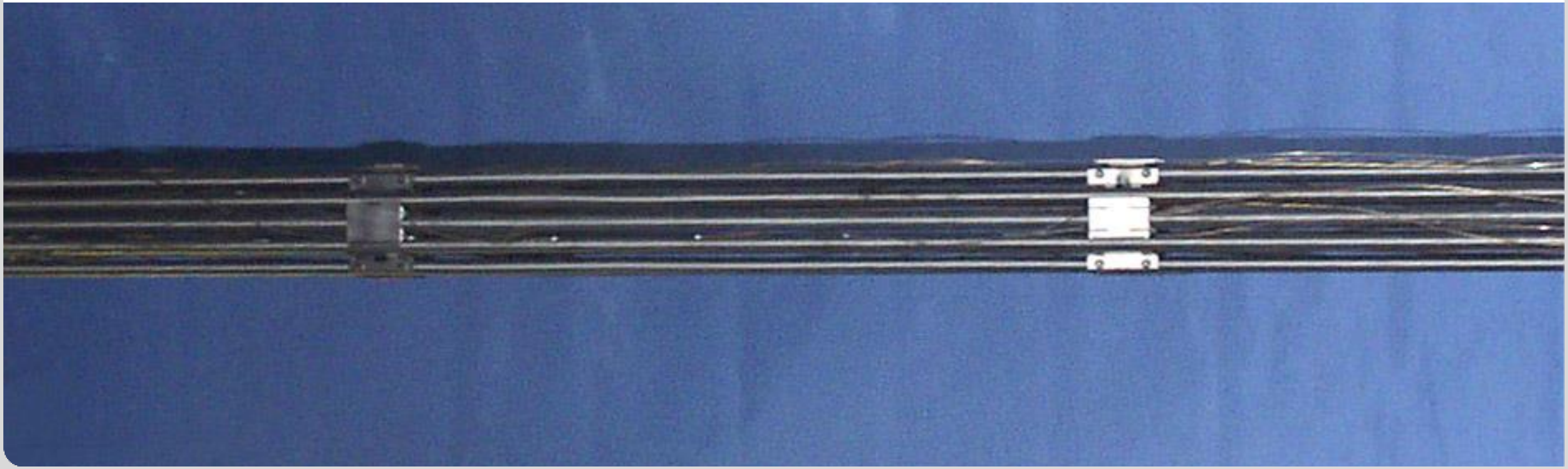


1. Preparation of samples for round robin tests;

- Cr coating of Zr-bearing claddings at OERLIKON BALZERS GmbH

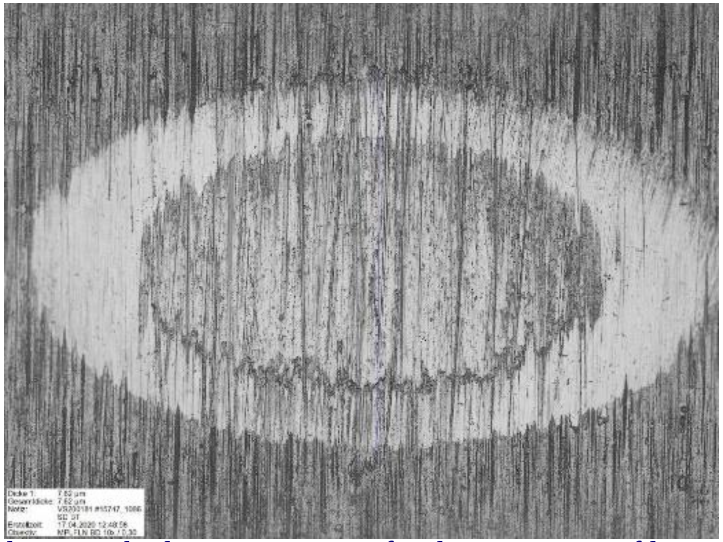
2. Two types of SETs at KIT at T=1200…1400 °C

- Long term oxidation tests inside tube furnace with heat insulation; common duration (transient + constant T) t > 500 s

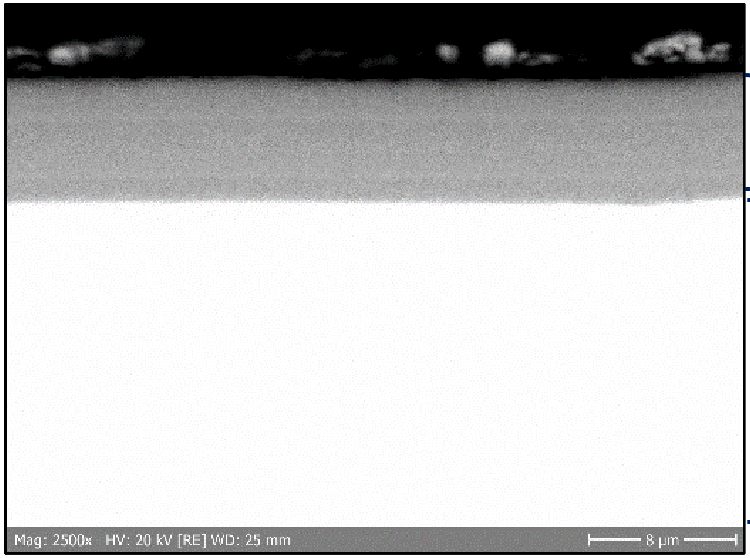
- Oxidation tests under LOCA conditions inside inductive furnace with radiative heat loss; common duration t < 500 s

Zr-alloy sample with PVD Cr coating with a layer thickness of 7 µm

Zr-alloy tube before coating by Cr at OERLIKON



Spherical cap method at OERLIKON for determination of layer thickness



Cr layer (3 sublayers)

Zr based substrate

SEM image of three PVD Cr layers

capillary Zr tube (sample holder and pressure normalizer)

Zr plug

Zr alloy cladding coated by Cr

Zr plug

coated empty sample welded at KIT and prepared for SETs

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Preparation and coating of samples for RRT

- 5 Zry-4 coated samples (length 130 mm, OD 10.75 mm, clad thickness 725 µm, Cr layer 7 µm) and corresponding ZrO_2 pellets sent to EK for ballooning tests (May 2020)
- 5 ZIRLO coated samples (length 130 mm, OD 10.75 mm, clad thickness 725 µm, Cr layer 7 µm) and corresponding ZrO_2 pellets sent to EK for ballooning tests (May 2020)
- 40 fresh Zry-4 samples (length 280 mm, OD 10.75 mm, thickness 725 µm) sent to CTU for coating (January 2021)
- 5 FeCrAl samples (ORNL B136Y3material, length 130 mm, OD 9.75 mm, clad thickness 380 µm) sent to EK for ballooning tests (February 2021)
- 11 fresh and 20 coated Zry-4 samples (length 280 mm, OD 10.75 mm, thickness 725 µm, Cr layer 10 µm) sent to CRIEPI for DEGREE bundle tests (February 2021)

I. Long term oxidation tests in the tube furnace LORA

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tube furnace

sample
withdrawn
from the furnace

Matrix of long term oxidation tests with M5 and ZIRLO claddings

MC tubes: OD=10.75 mm, wall 725 μm;   MCs and Zos tubes: OD=9.5 mm, wall 570 μm

Sample	Atmosphere	Time to reach maximal temperature [min]	Maximal temperature [°C]	Duration of oxidation [min]	Cooldown	Comment
MC-1	O_2+Ar	-	setup 1345 escalation to 1787	-	-	performed; sample significantly melted
MC-2	O_2+Ar	30	1200	30	in air at RT	performed
MC-3	O_2+Ar	37.5	1300	10	in air at RT	performed
M-4	*O_2+Ar*	*30*	*1200*	*30*	*in air at RT*	*performed; sample not coated; reference test for MC-2*
MCs-1	O_2+Ar	41.25	1350	5	in air at RT	performed
Zos-1	O_2+Ar	41.25	1350 (escalation to 1500)	5	in air at RT	performed; sample partially melted

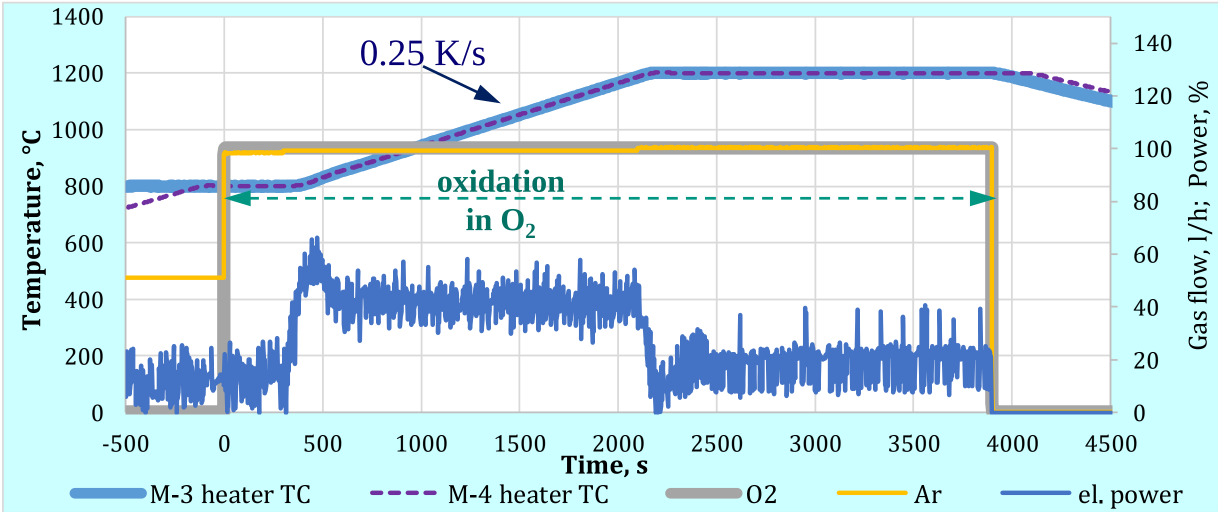
Appearance of coated cladding samples oxidized in tube furnace

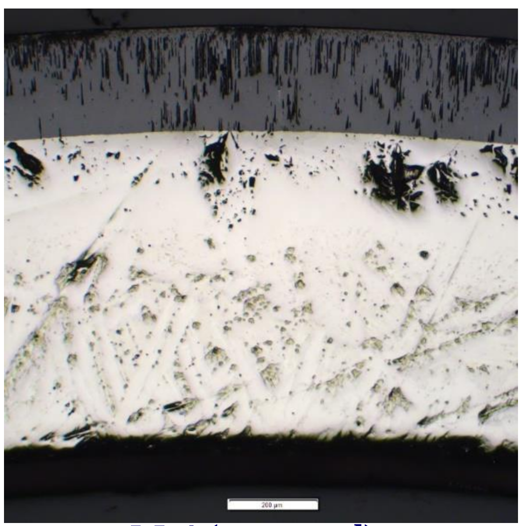
MC-2:
1200 °C
during 1800 s

MC-3:
1300 °C
during 600 s

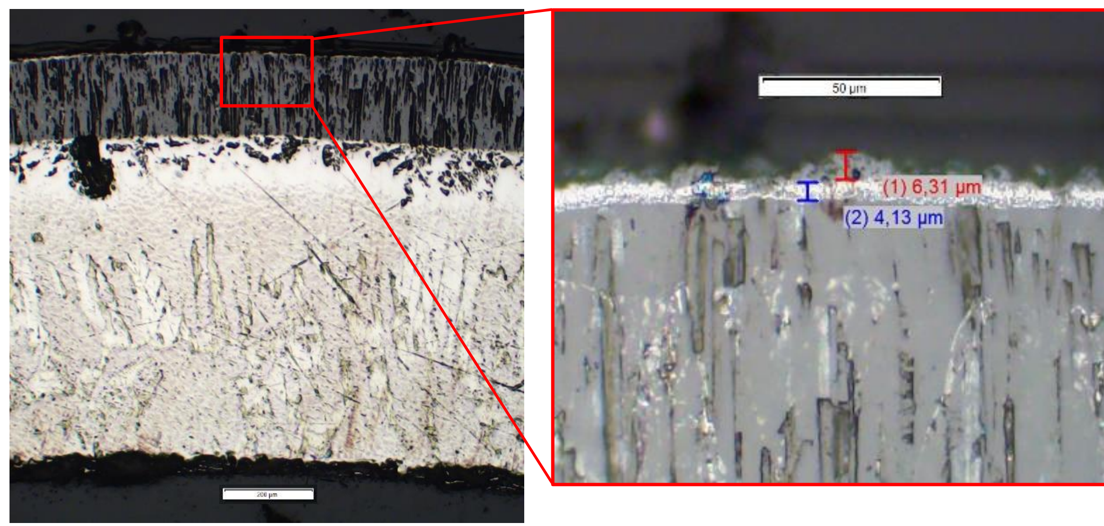
MCs-1:
1350 °C
during 300 s

Long term oxidation in O_2: comparison of uncoated M-4 sample with coated MC-2 sample





M-4 (uncoated):
ZrO_2 225 µm; α-Zr(O) 240 µm



MC-2 (coated):
ZrO_2 198 µm; α-Zr(O) 120 µm

Outer layers after long term oxidation

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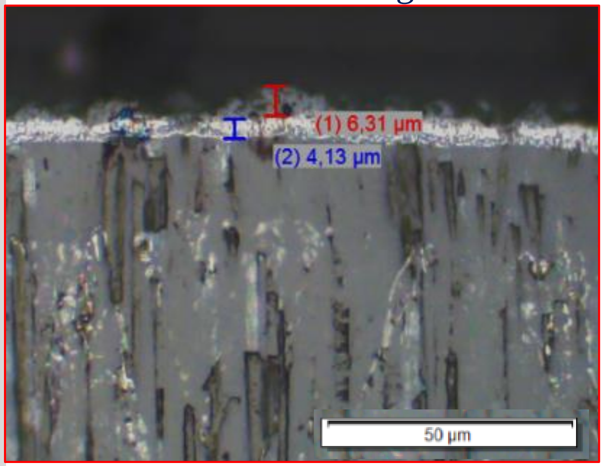
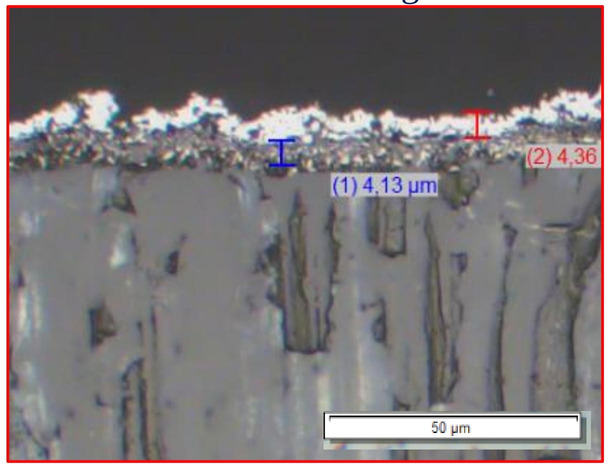
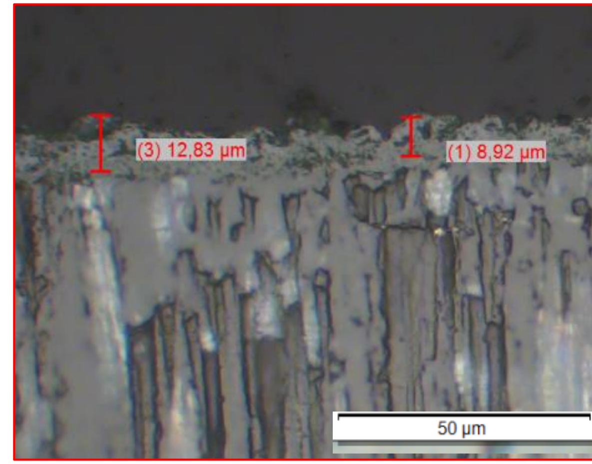
MC-2: 1200 °C during 1800 s

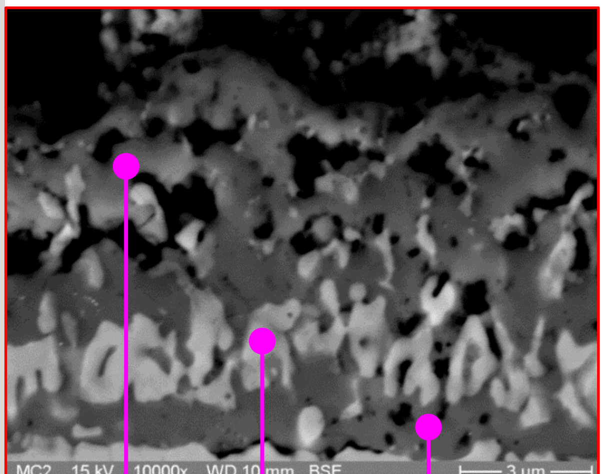


MC-3: 1300 °C during 600 s

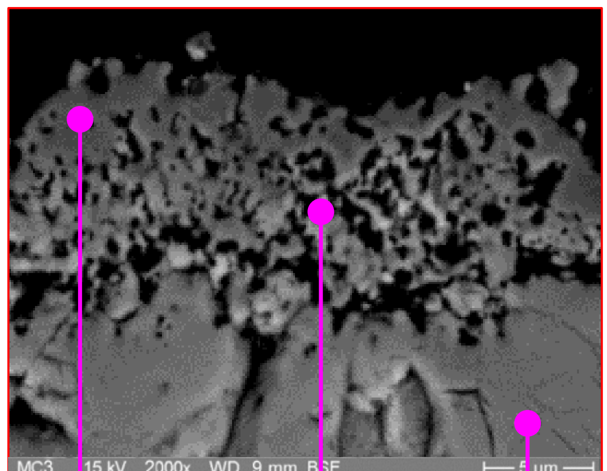


MCs-1: 1350 °C during 300 s

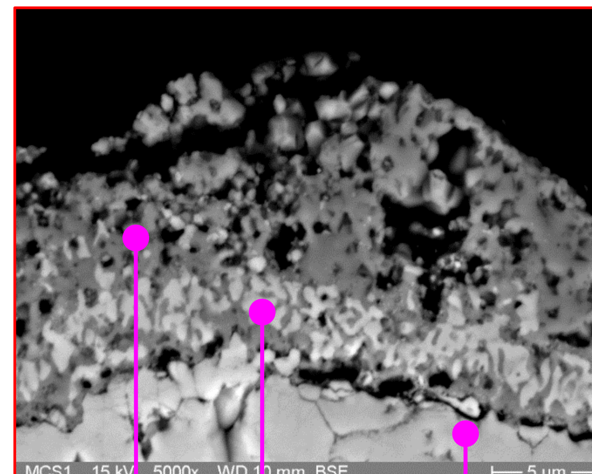




Cr_2O_3 α-Zr(O) +Cr Cr_2O_3



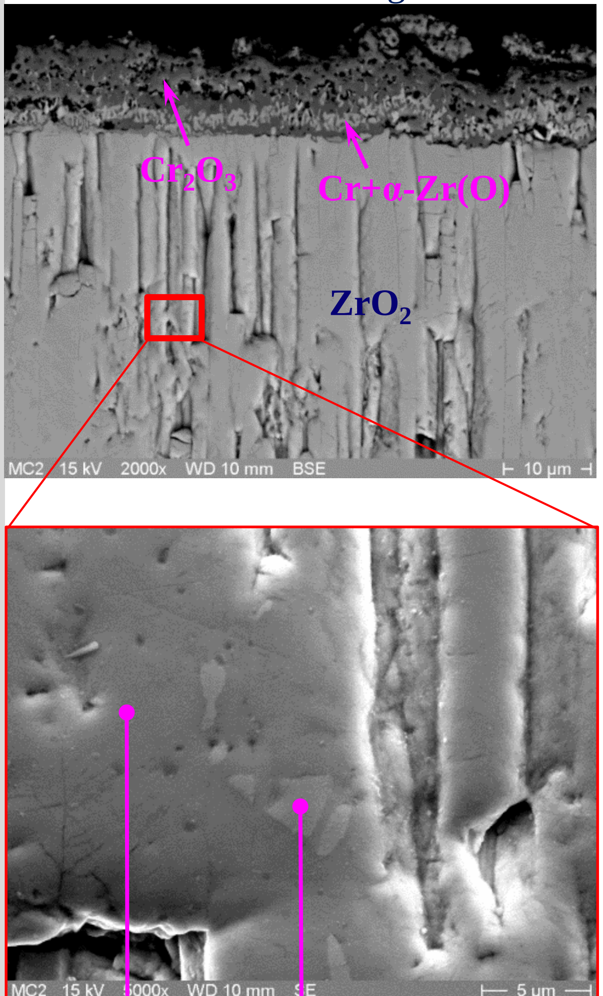
Cr $+Cr_2O_3$ Cr_2O_3 $+ZrO_2$ ZrO_2



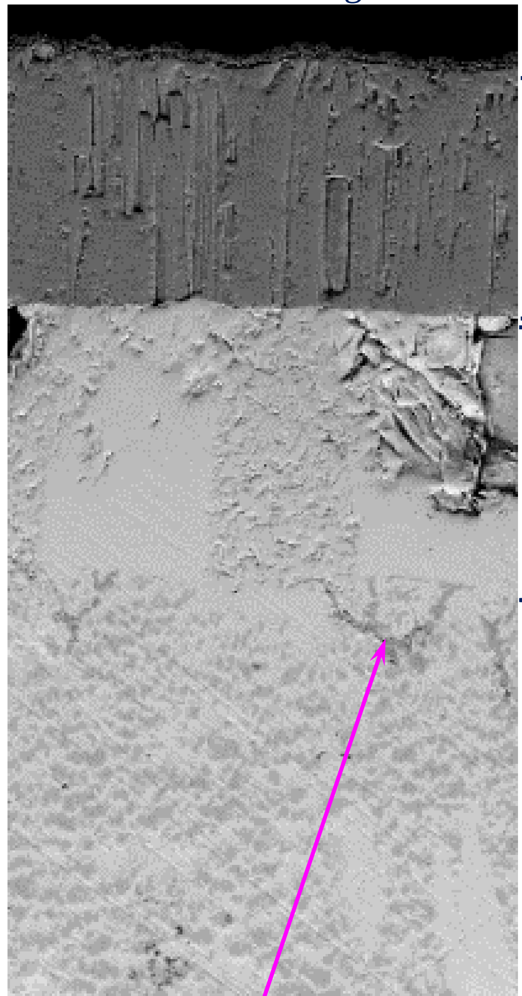
Cr_2O_3 α-Zr(O) +Cr ZrO_2

Cr diffusion into the cladding bulk during long term oxidation

MC-2: 1200 °C during 30 min

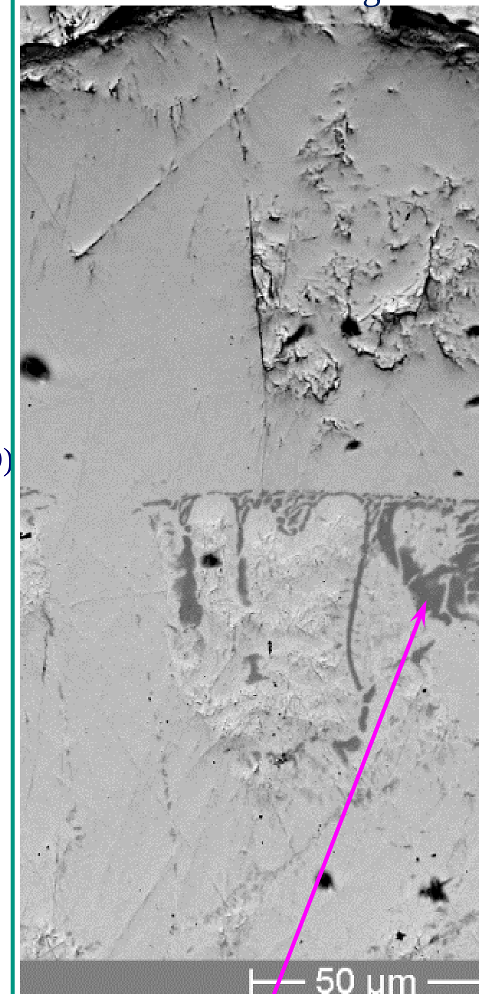


MC-3: 1300 °C during 10 min



Cr precipitates at the boundary between α-Zr(O) and β-Zr

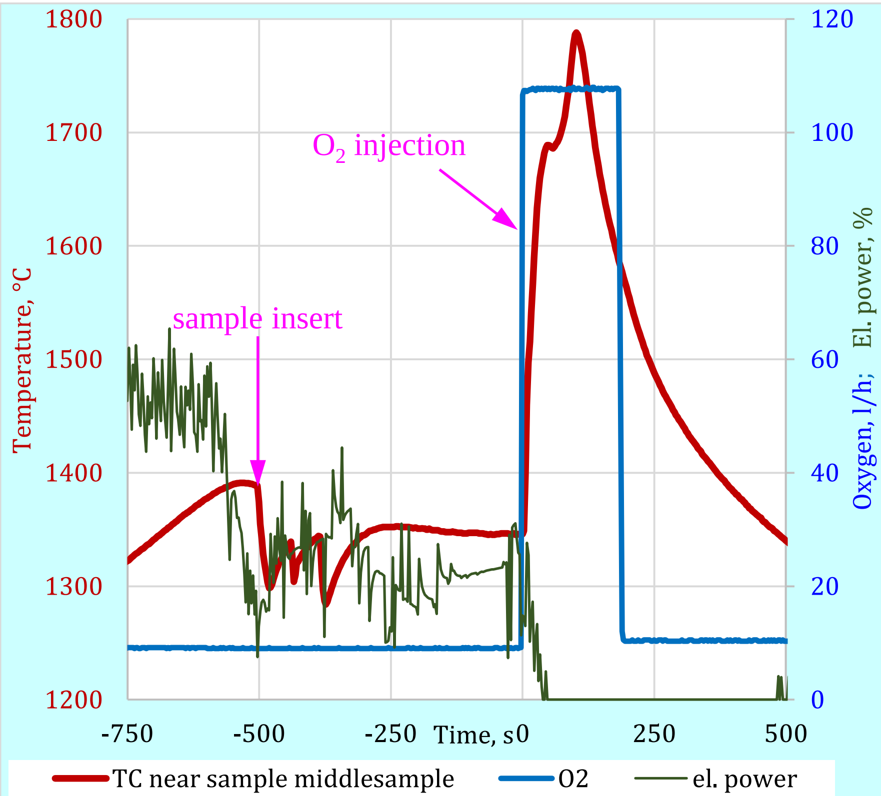
MCs-1: 1350 °C during 5 min



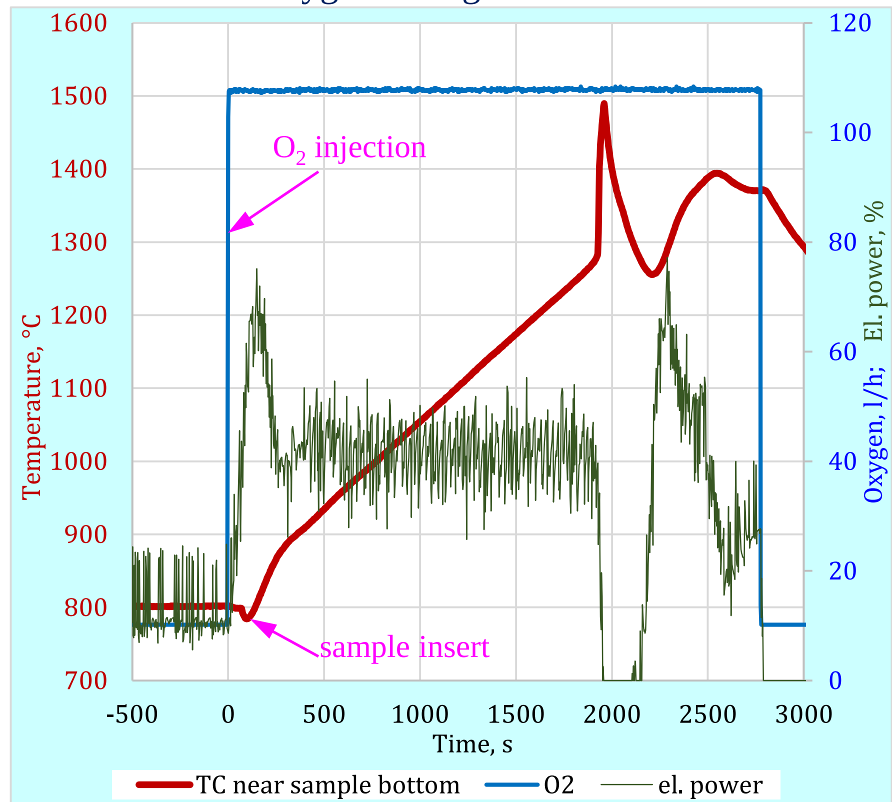
Cr precipitates at the boundary between α-Zr(O) and β-Zr

Catastrophic oxidation and temperature escalation at T>1300 °C in tube furnace (without radiation heat loss)

sample MC-1: injection of oxygen at 1350°C



sample ZOs-1: temperature escalation in oxygen during slow transient

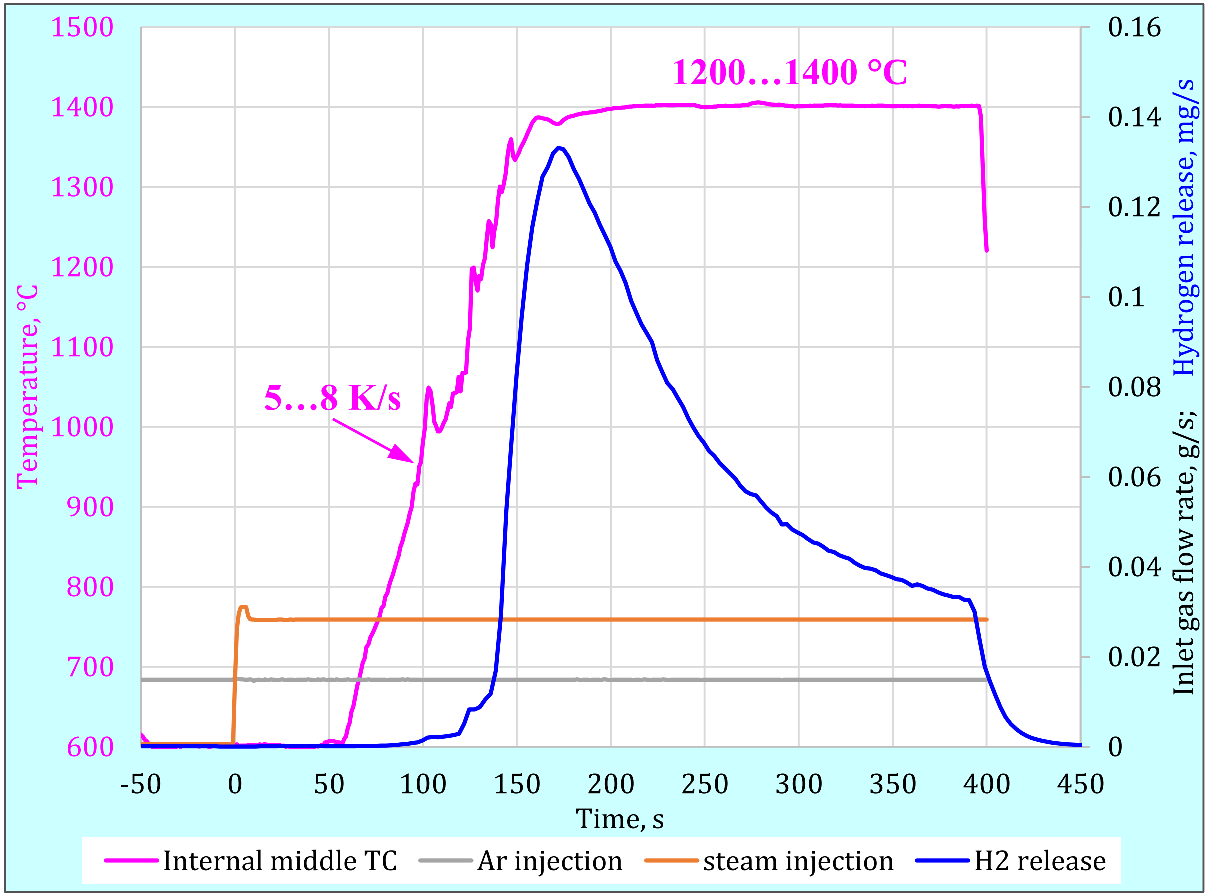


sample completely destroyed

II. Simulation of LOCA heating rates in inductive furnace: cladding oxidation in steam

H_2+Ar + steam

Ar + steam



Matrix of single rod tests to perform under LOCA conditions

MC tubes: OD=10.75 mm, wall 725 µm;   MCs and Zos tubes: OD=9.5 mm, wall 570 µm

Sample	Heating rate [K/s]	Maximal clad temperature T_{max} [°C]	Duration of oxidation at T_{max} [min]	Cooldown	Comment
MC-4	7	1200	10	steam + Ar	performed
MC-5	7	1200	15	steam + Ar	performed
MC-6	8	1300	5	steam + Ar	performed
MC-7	6	1250	10	steam + Ar	performed
MCs-2	8	1360	6	steam + Ar	performed
MCs-3	8	1360	0	steam + Ar	performed
MCs-4	5	1250	6 (1250°→900°)	water	performed
MCs-5	5	1250	0	steam + Ar	performed
ZOs-2	5	1200	4	steam + Ar	performed
ZOs-3	5	1380	0	steam + Ar	performed
ZOs-4	5	1200	4	steam + Ar	performed
ZOs-5	5	1400	4	steam + Ar	performed

Appearance of coated cladding samples oxidized in inductive furnace

MC-4: 1200 °C
during 10 min

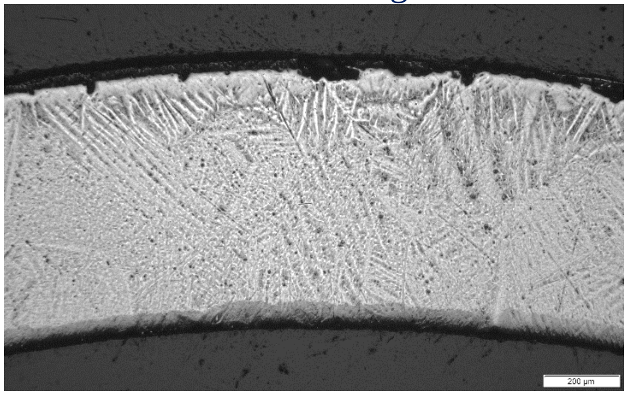
MC-5: 1200 °C
during 15 min

MC-7: 1250 °C
during 10 min

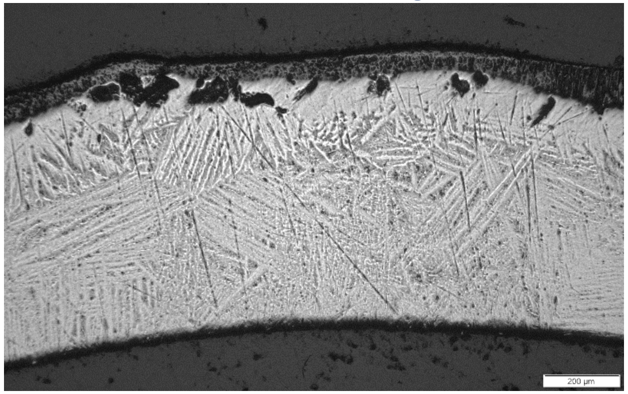
MC-6: 1350 °C
during 5 min

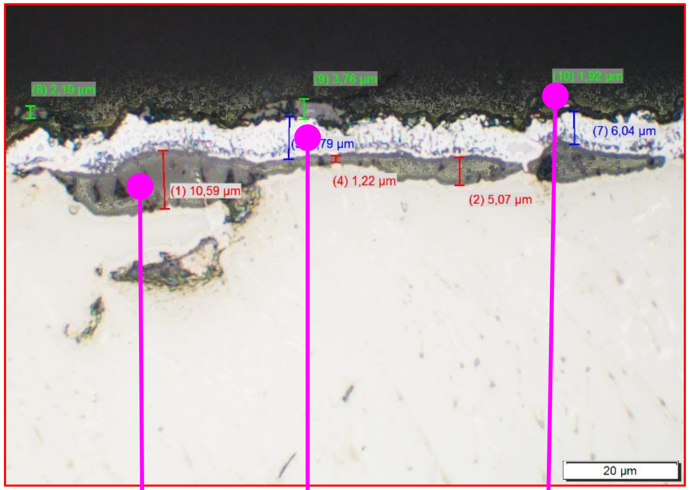
LOCA heating rate: cladding microstructure after oxidation at 1200 °C

MC-4: 1200 °C during 10 min



MC-5: 1200 °C during 15 min





ZrO_2: 2…25 µm

Cr: 5µm

Cr_2O_3: 5µm

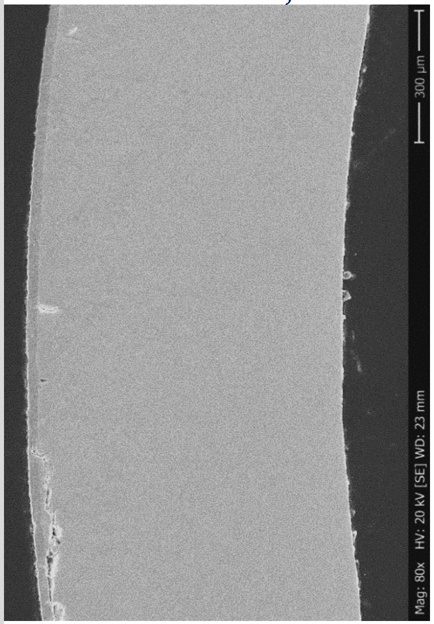
ZrO_2: 75 µm

Cr_2O_3: 3…7 µm

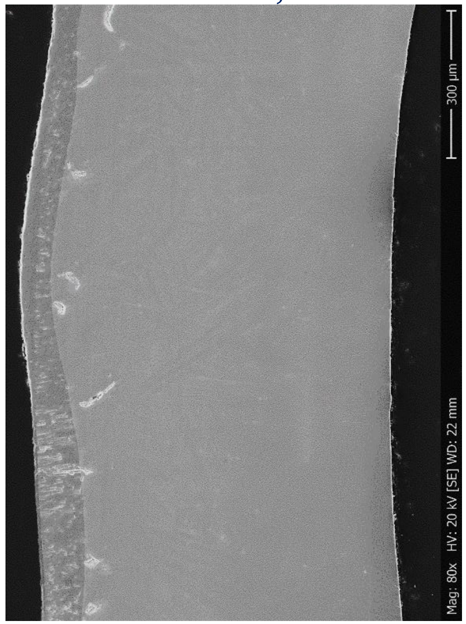
Cr: 0…7 µm

LOCA heating rate: diffusion of Cr through the cladding layers

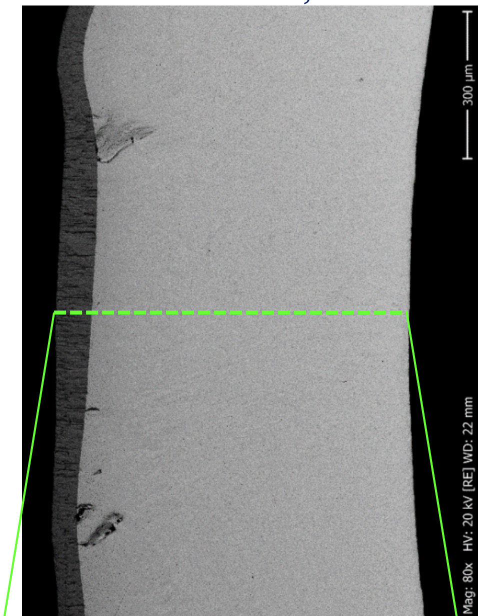
MC-4: 1200 °C;10 min

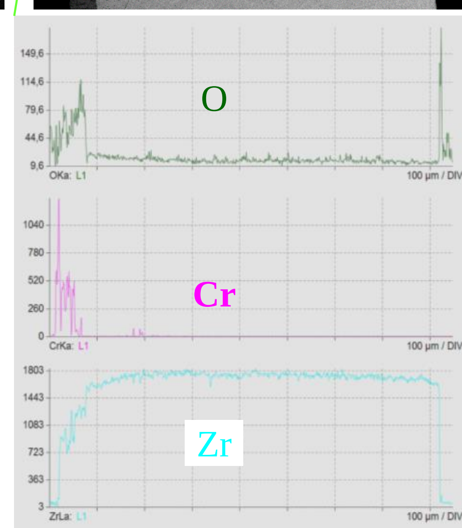


MC-5: 1200 °C; 15 min

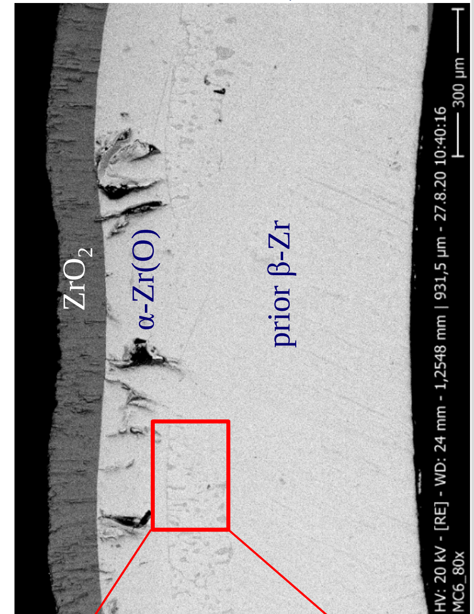


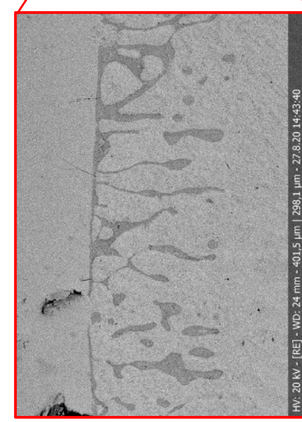
MC-7: 1250 °C; 10 min





MC-6: 1350 °C; 5 min





Cr precipitates at the boundary between α-Zr(O) and β-Zr

LOCA transient followed by a constant temperature of 1400 °C: influence of duration of oxidation in steam

sample MCs-3: transient with 8 K/s, then 1400 °C during 3 s

sample MCs-2: transient with 8 K/s, then 1400 °C during 240 s

1400 °C: deformation of outer surface

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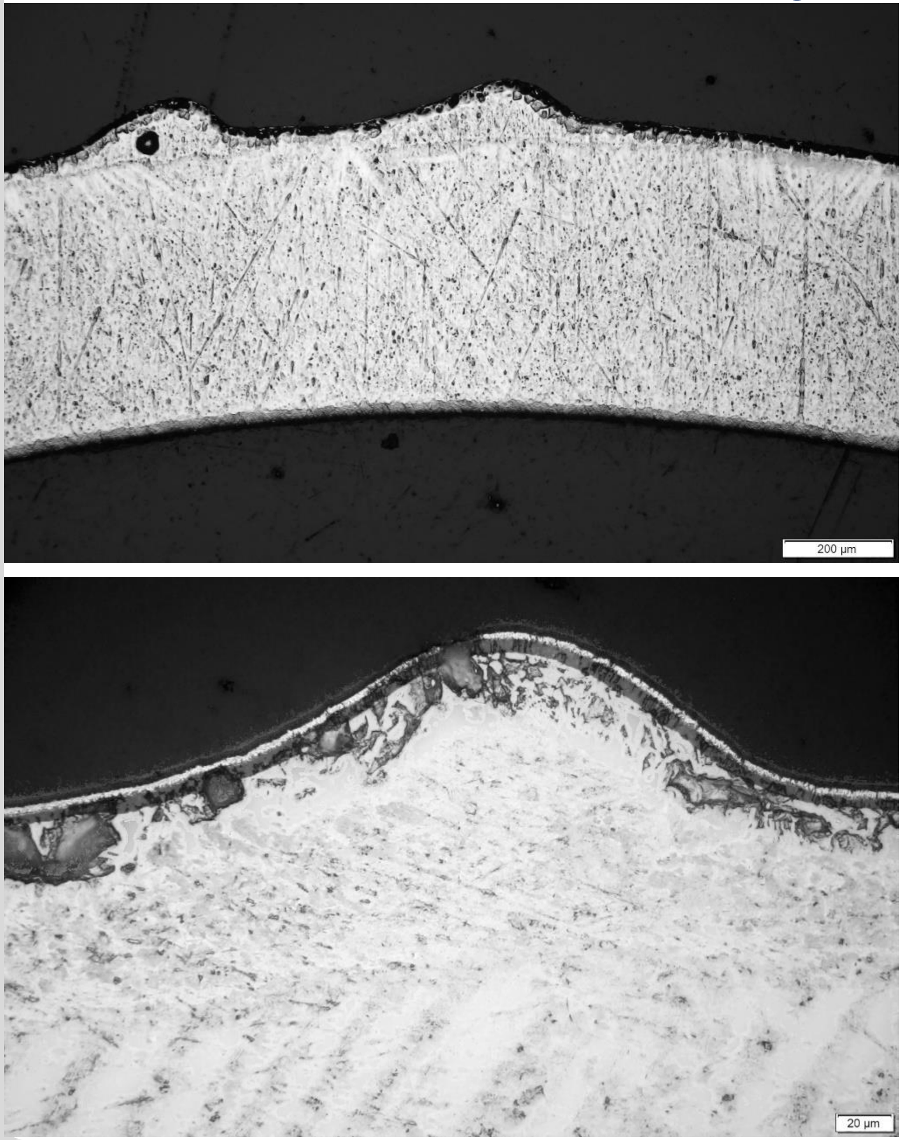
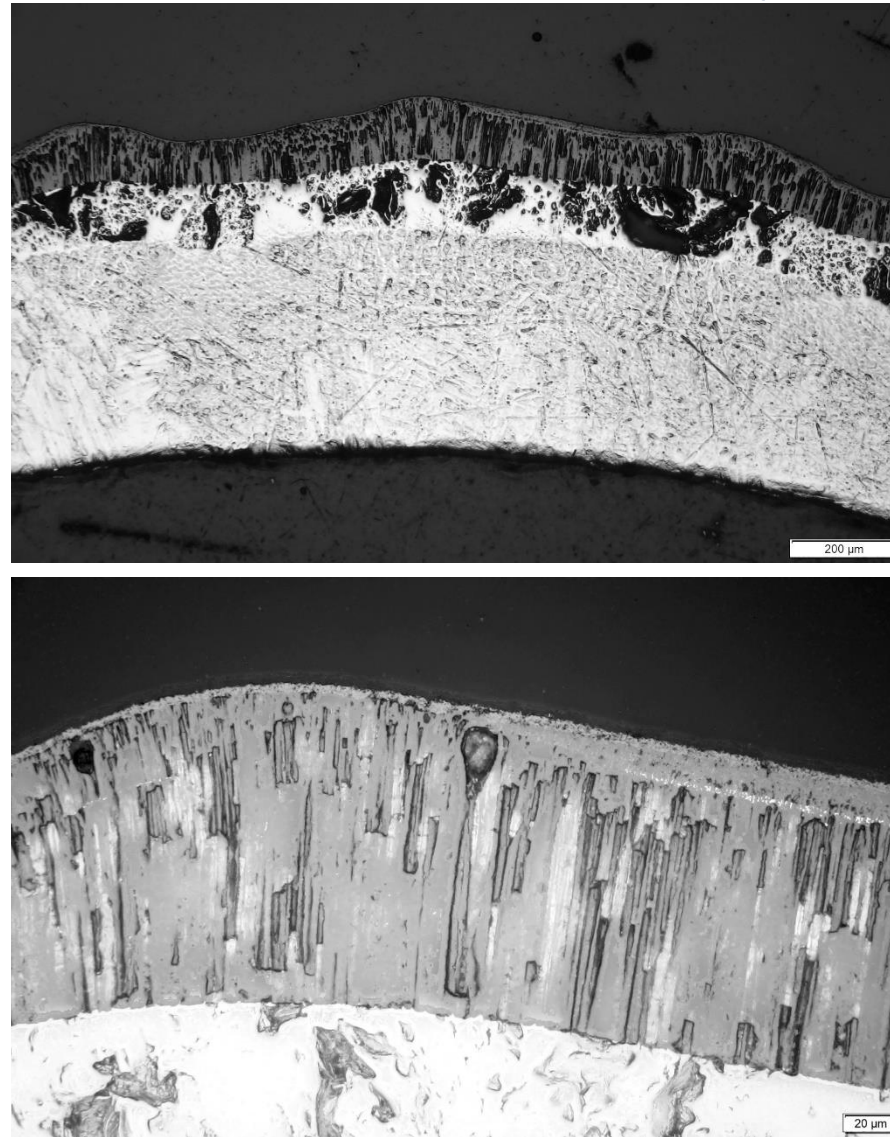
MCs-3: transient with 8 K/s, then 1400 °C during **3 s**



MCs-2: transient with 8 K/s, then 1400 °C during **240 s**



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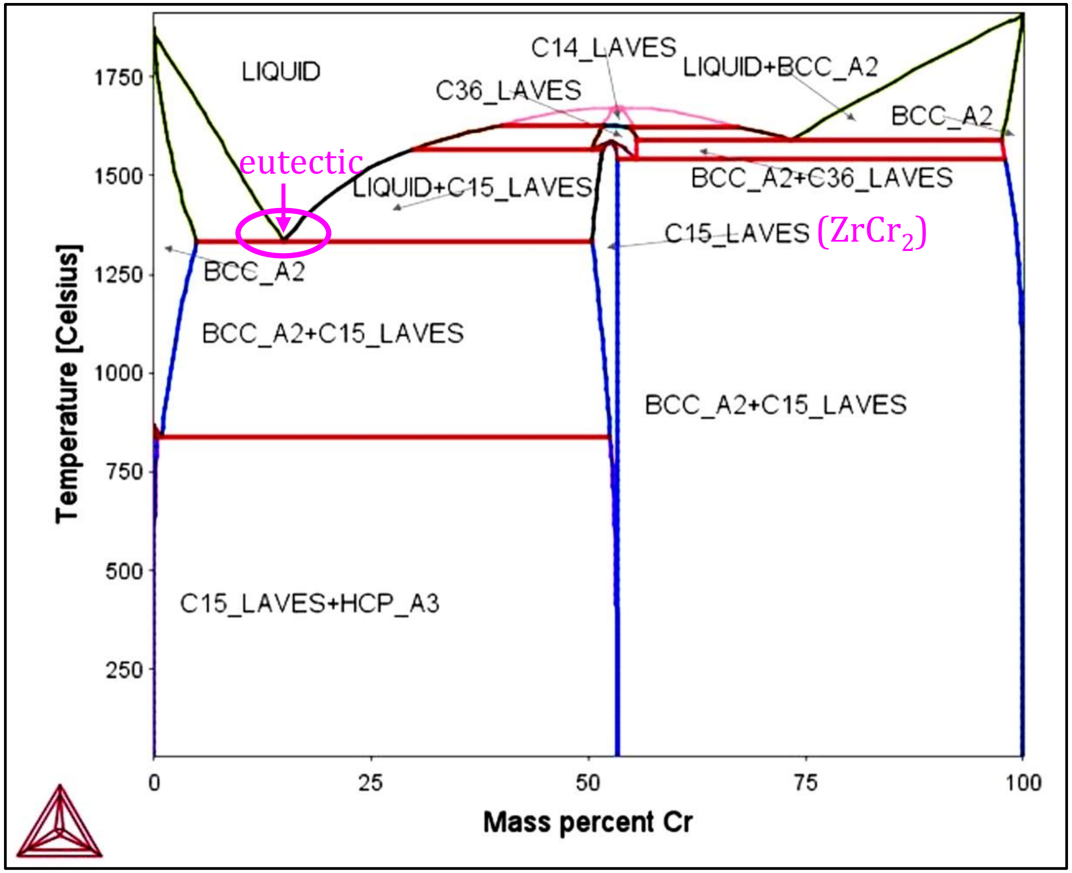
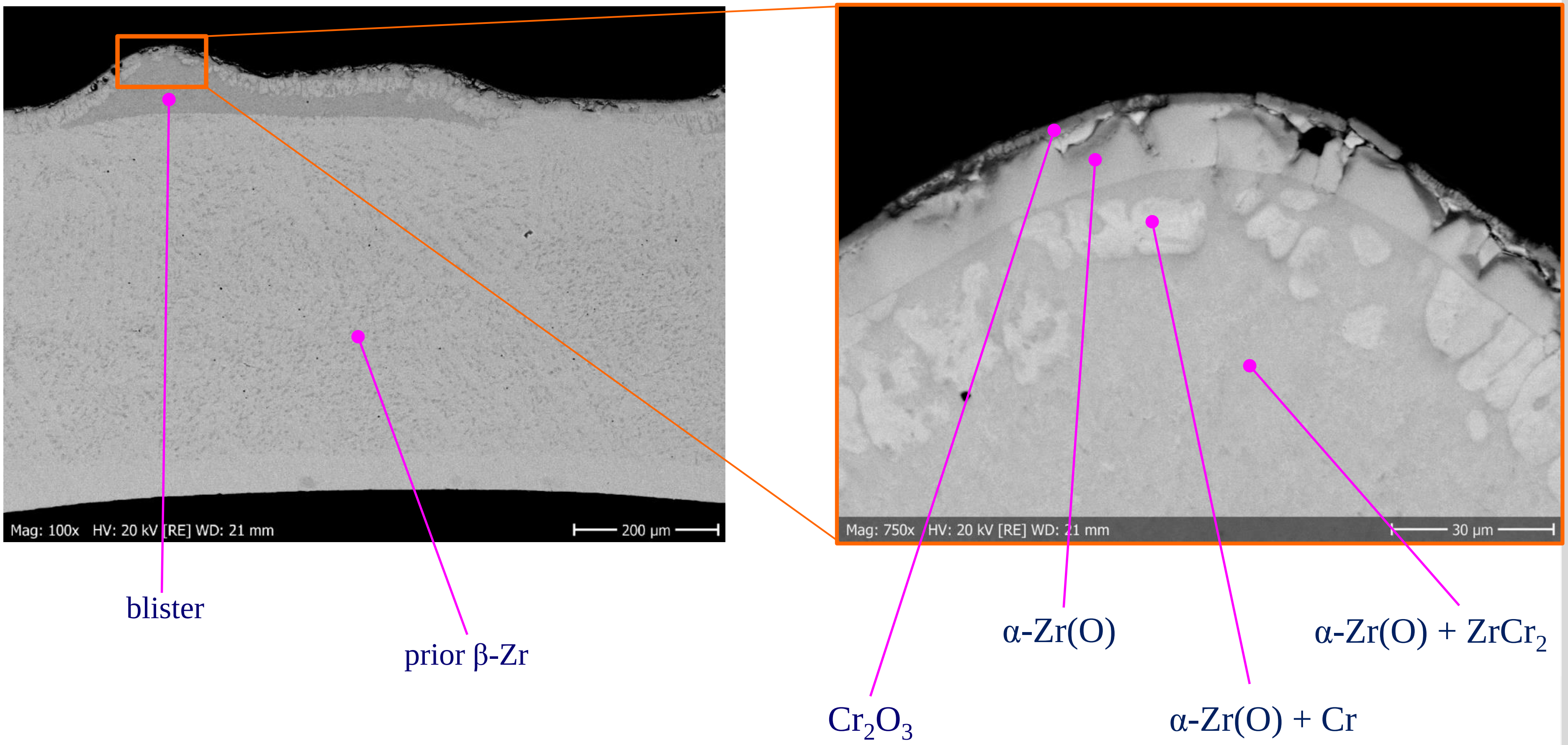
Binary Zr-Cr phase diagram

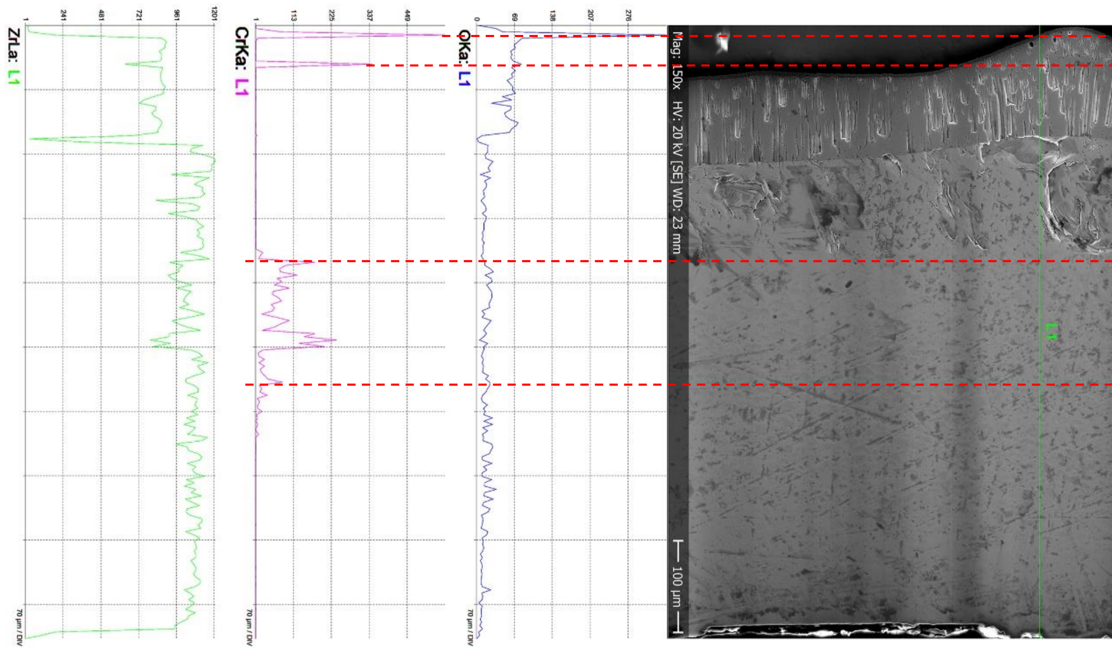


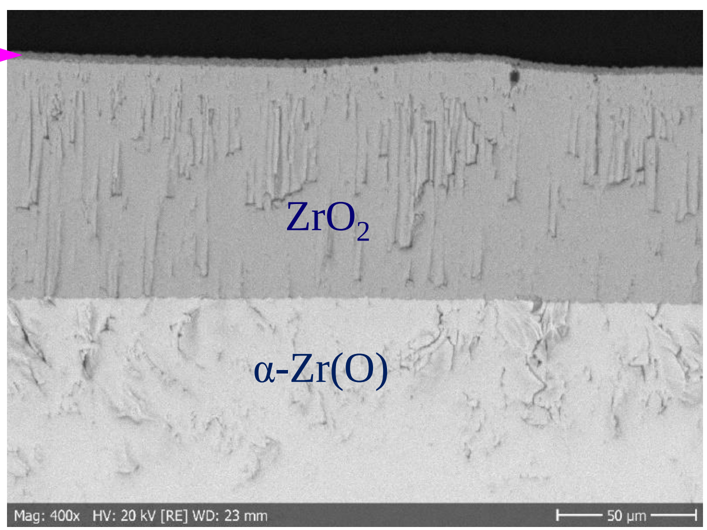
The eutectic forms at ≈1330°C

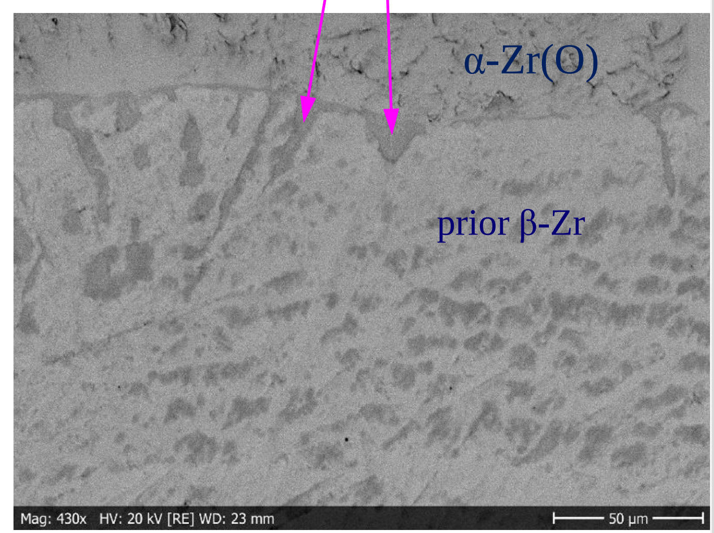
Result of SEM/EDX analysis: cladding microstructure immediately after LOCA transient from 600 to 1400 °C



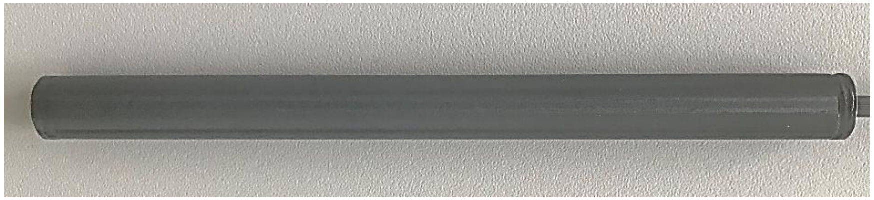
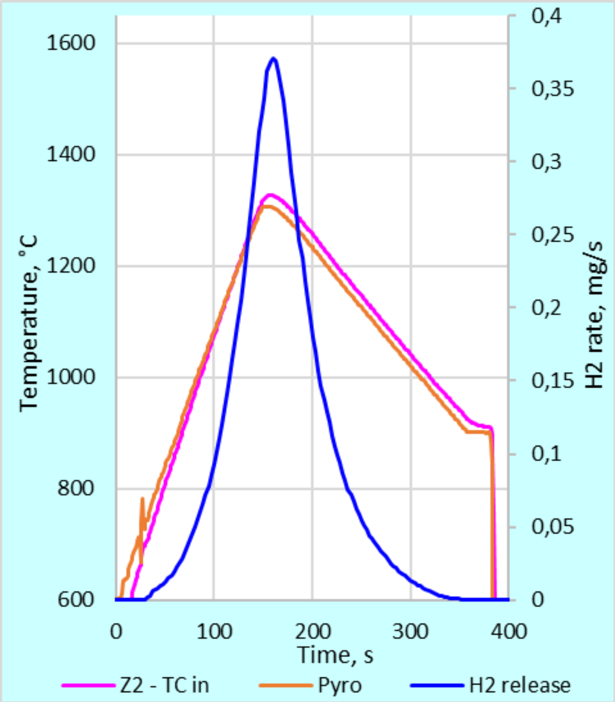
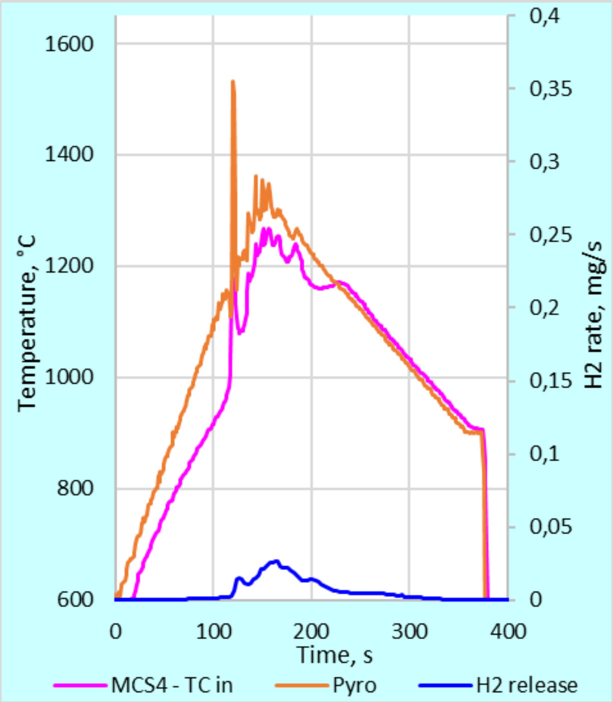
Result of SEM/EDX analysis: cladding microstructure after LOCA transient and following holding at 1400 °C during 240 s



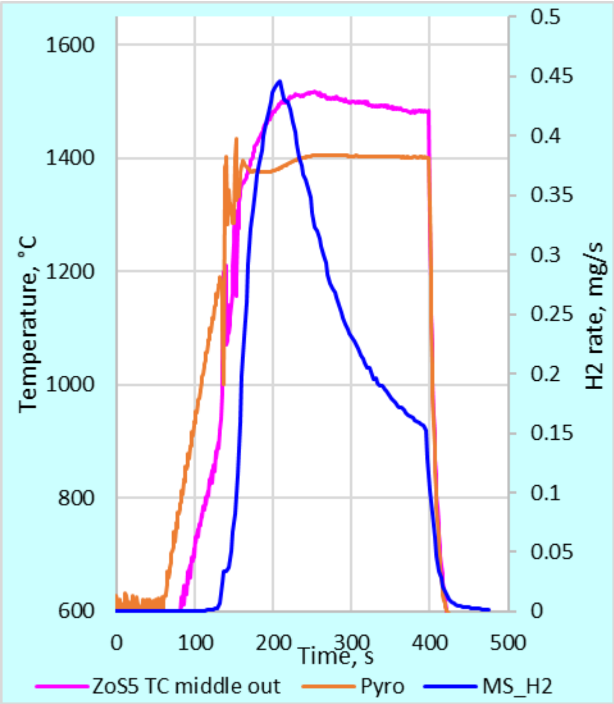


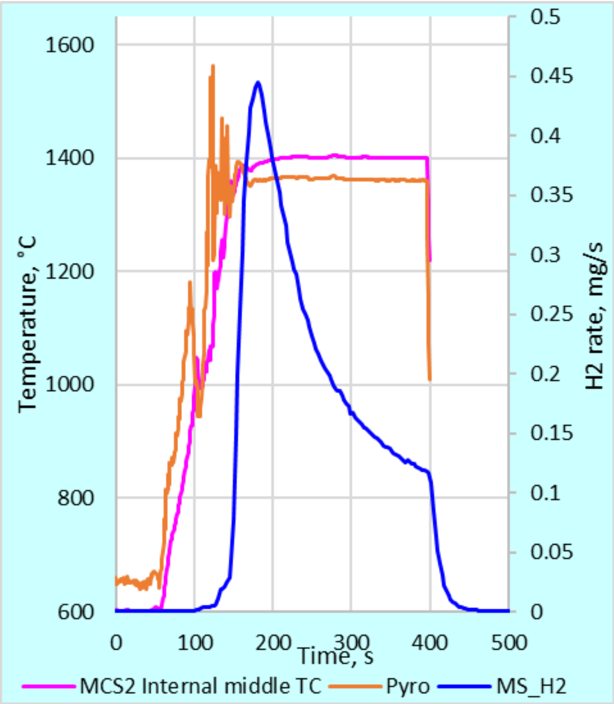


Influence of coating on hydrogen release
(LOCA transient 5 K/s from 600 to1250 °C, then cooldown to 900 °C and quenching)

	
 	
not coated sample Z2 (Zry-4): significant hydrogen release	coated sample MCs-4 (M5): strong reduced hydrogen release (factor 15)

Influence of Zr alloy (as substrate) on hydrogen release for coated samples tested under similar temperature conditions





sample Zos5 (ZIRLO)	sample MCs2 (M5): decreased hydrogen release (factor 4)

Summary and conclusions

- Low heat-up rate: decrease of cladding surface oxidation for Cr coated samples in comparison to not coated samples; moderate decrease of ZrO_2 growth, significant decrease of α-Zr(O) growth.
- Catastrophic oxidation at T>1300 °C in the absence of radiation heat loss.
- Diffusion of Cr through ZrO_2 and α-Zr(O) layer and Cr precipitation at the boundary between α-Zr(O) and prior β-Zr layers.
- Fast (LOCA) transient: numerous blisters (local swellings) at the outer cladding surface. Zr-Cr eutectic at 1350 °C and formation of Laves phase $ZrCr_2$.
- Influence of direct heating on blister formation: hot spots due to higher electrical conductivity of Cr compared to Zr (factor 3)?

Thank you for your attention

http://www.iam.kit.edu/awp/163.php
http://quench.forschung.kit.edu/