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Structrual Materials Development and Heavy Liquid Metal Technology for Advanced Nuclear Reactors

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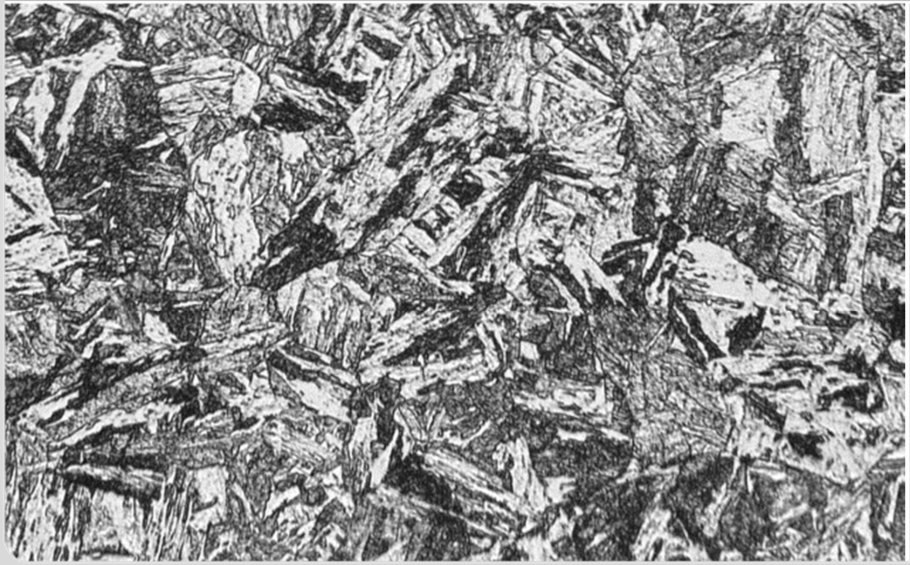
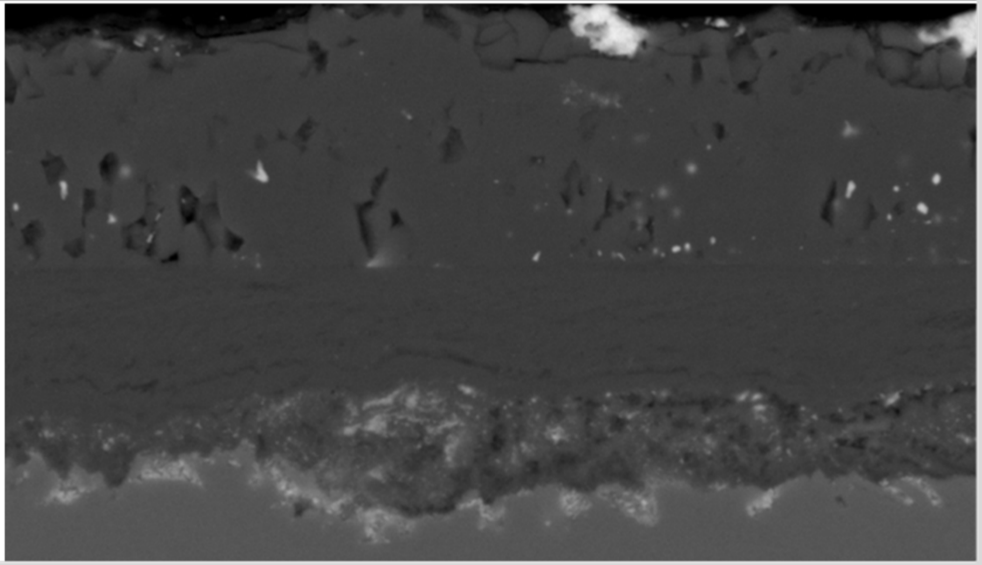
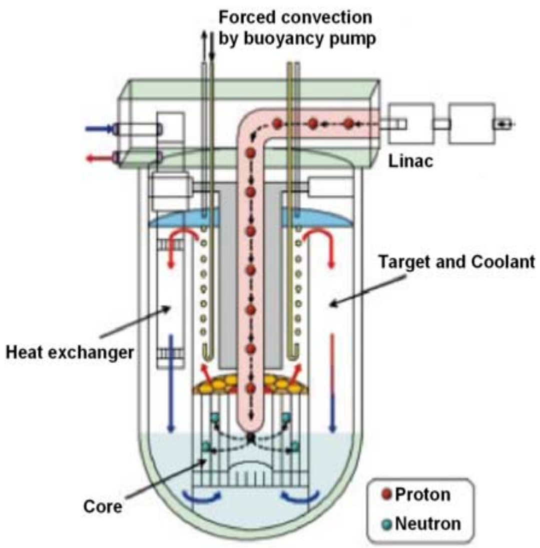
INSTITUTE FOR APPLIED MATERIALS – MATERIAL PROCESS TECHNOLOGY (IAM-WPT)





Lead-cooled Nuclear Reactors/Systems

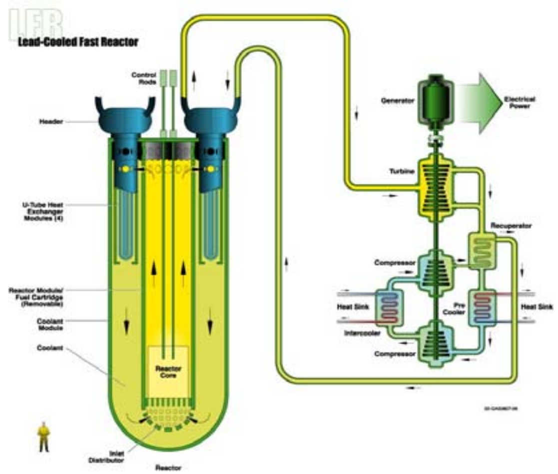


Accelerator Driven (Subcritical) System

- Transmutation of long-lived radioactive isotopes in nuclear waste
- Power generation
- Liquid lead (Pb) or lead-bismuth eutectic (LBE) as spallation target and primary coolant
- Maximum temperature, typically
  - 450 – 500°C for regular operation
  - Periodically 550°C (according to plant design)

Lead-Cooled Fast Reactor

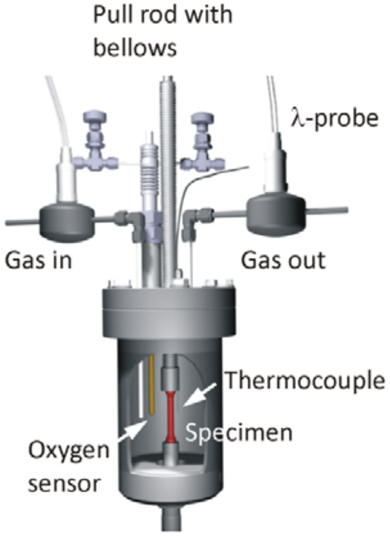
- One of the concepts for the 4th generation of nuclear power plants (Gen IV)
- In the long-term, Pb as primary coolant at maximum ca. 800°C
- Short- to mid-term: Pb- or LBE-cooled at 450 – 550°C



Activities at KIT / IAM-WPT related to ADS and LFR

- Long-term corrosion in oxygen-containing lead-bismuth eutectic (LBE) and Pb
  - At a flow velocity of 2 m/s
  - T = 450–550°C
  - $c_O = 10^{-7}–10^{-6}$ mass%

- Creep-to-rupture in oxygen-containing Pb alloys
  - Static Pb or LBE
  - T = 450–650°C
  - $c_O = 10^{-7}–10^{-6}$ mass%



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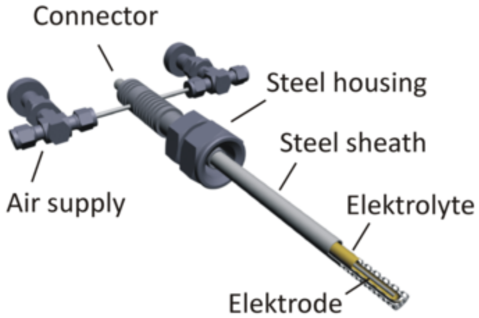
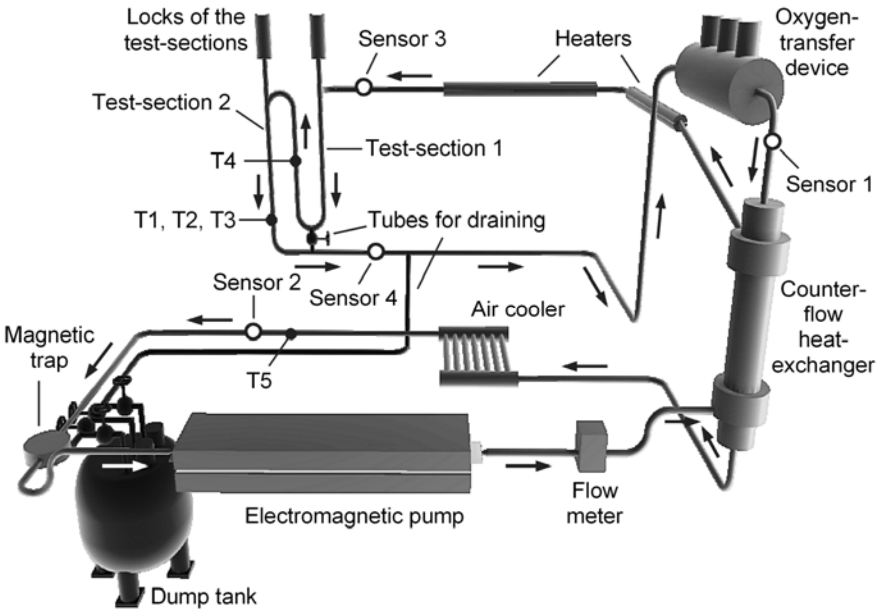


- Instruments and methods of oxygen control in Pb alloys
  - Via oxygen-containing gas
  - (gas/liquid oxygen transfer)
  - Oxygen sensors

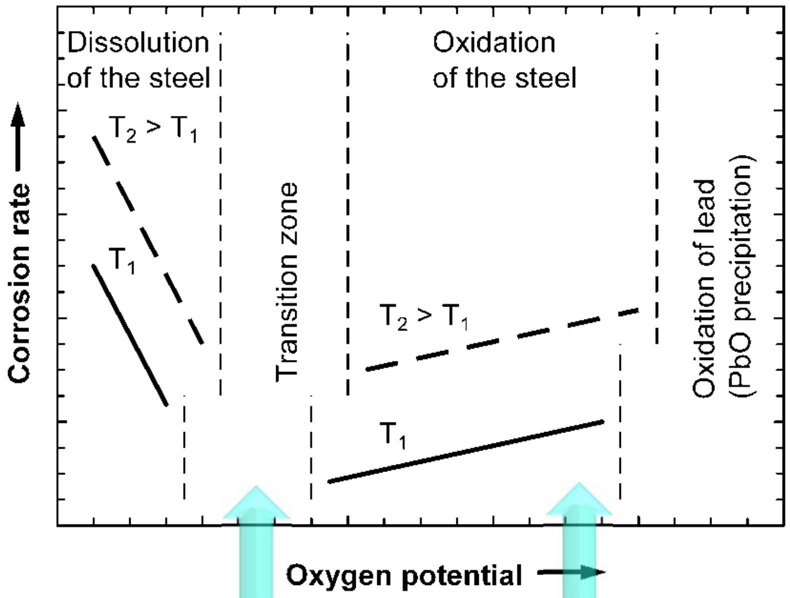
CORRIDA: Corrosion-testing in dynamic lead alloys

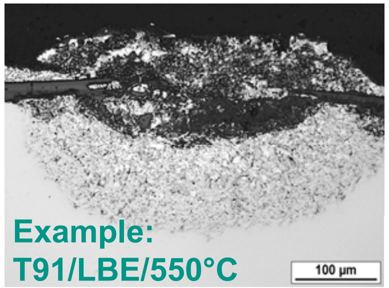


- **Technical data**
  - Material: SS 316-Ti (DIN 1.4571)
  - Developed length: 36 m
  - Liquid metal: ~1000 kg LBE
  - Mass flow: 5.3 kg/s (steady state)
  - T_{max} = 550°C (test-sections, oxygen control-box)
  - T_{min} = 350–385°C depending on T_{max} at inlet of EM-pump
  - Oxygen control:
  - Gas with adjustable O_2-content introduced at T_{max}

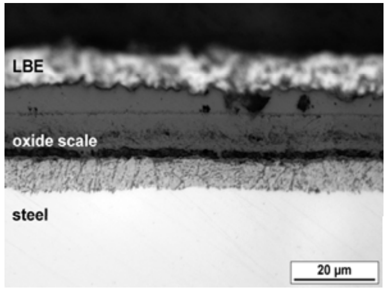
- **Operating data**
  - Commissioning in July 2003
  - ca. 70,000 h of effective operation at Tmax = 450 to 550°C
  - Longest exposure time of specimens: 20,000 h

Impact of oxygen addition to Pb alloys on steel corrosion





Example: T91/LBE/550°C



- **Stimulation of the oxidation of steel constituents**
  - Formation of an oxide scale on the steel surface
  - Spatial separation of the steel from liquid metal
  - Reduced dissolution rate or risk of embrittlement
- **Steel constituents must be less noble than the constituents of the liquid metal**
  - Applicable to Pb, lead-bismuth
  - Not applicable to lead-lithium (Pb17Li) or Na
- **However, thick oxide scales impair heat-transfer across the steel surface**
  - Practical limit of oxygen addition
- **Relevant to**
  - Lead-cooled fast reactor (LFR)Accelerator driven system ("Actinide Burner")

Components of an oxygen control system

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Sensors for on-line monitoring

Electrochemical oxygen monitoring

- Solid electrolyte on the basis of yttria-stabilized zirconia (YSZ)
- Metal/metal-oxide or Pt/gas reference electrode

Issues to be addressed (in general)

- Compatibility with the use in Pb alloys (YSZ/steel joint)
- Accuracy
- Long-term reliability

Licensing for nuclear application

- Structural stability of the YSZ product used
- Risk of contamination in case of electrolyte cracking

Oxygen-transfer device(s)

"Classic" mass transfer across the interface between oxygen source/sink and the liquid metal

Type	Oxygen source	Oxygen sink
Solid-liquid	PbO	(less noble metals)
Gas-liquid	Ar, H_2O, air	Ar-H_2

Long-term experience from operating experimental facilities for testing materials (steels) in oxygen-containing Pb alloys exists

Electrochemical oxygen sensors for liquid lead alloys

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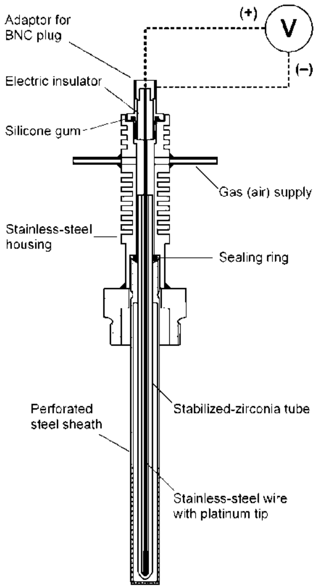
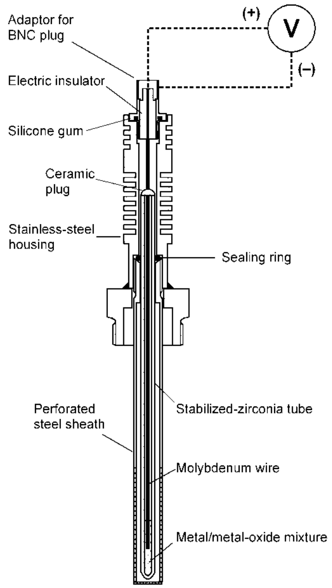
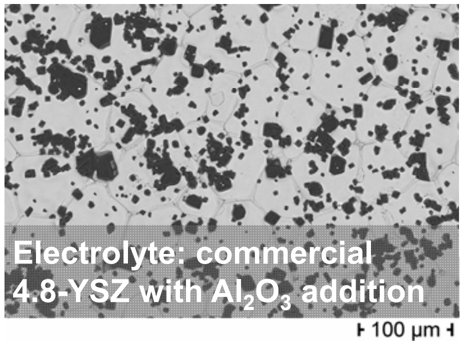
Basic components	Sensor output
▪ Solid electrolyte ▪ Yttria stabilized zirconia (YSZ) ▪ Tubes with 4.5–4.8 mole% Y_2O_3 ▪ "Thimble" with 3 mole% Y_2O_3	▪ Voltmeter reading, E ▪ Measure of the chemical potential of oxygen in the liquid metal ▪ May in general depend on the specific combination of the sensor with a high-impedance voltmeter
▪ Reference electrode ▪ Metal/metal-oxide like Bi/Bi_2O_3 and In/In_2O_3 with Mo wire as electric lead ▪ Pt/air using steel wire with platinised tip as electric lead	▪ Ideal sensor/voltmeter system ▪ Ideal zero-current potential: $E^* = \frac{(\mu_{O_2;Ref} - \mu_{O_2})}{4F}$
▪ Second (working) electrode ▪ The liquid Pb alloy ▪ Auxiliary wire or the steel housing of the sensor serves as part of the electric lead	▪ Calculated oxygen concentration, c_O: $\log(c_O / \text{mass\%}) = C_1 + \frac{C_2}{T/K} - 10{,}080 \frac{E^*/V}{T/K}$ ▪ C1 and C2 are constants specific for the reference electrode

Oxygen sensors developed at KIT

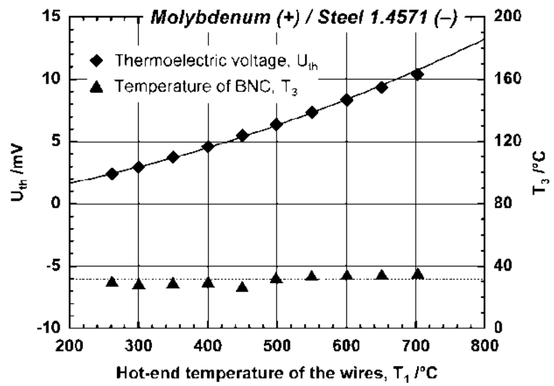


- Long electrolyte tube (Ø 6×255 mm)
- Polymer sealing ring in sufficient distance from the liquid metal
- Cooling fins for reducing the thermal load on the sealing ring
- Steel sheath for protecting the electrolyte from shear forces, serving as electric lead on the liquid-metal side
- Reference electrodes
  - (Steel)Pt/air
  - (Mo)Bi/Bi_2O_3



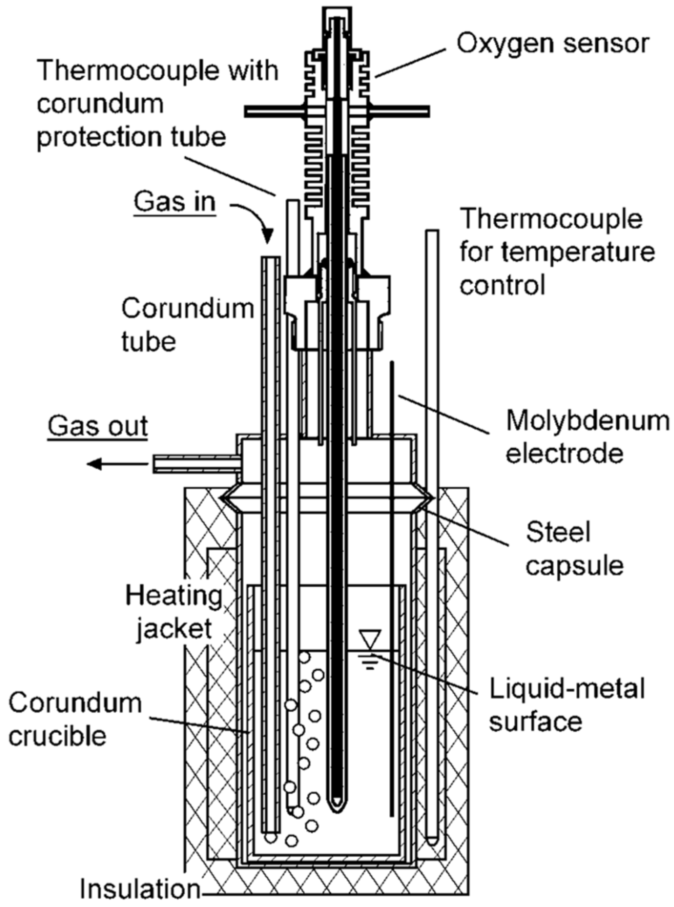


U_{th} :
~3 mV at 300°C
~11 mV at 700°C (Mo/stainless steel)



Testing of the sensor accuracy

Adjusting known oxygen potentials in LBE
Pb/PbO (oxygen saturation)
Co/CoO
Fe/Fe-oxide equilibria
Fe and Co added in the form of powder
Stabilization of these potentials using gases with varying oxygen partial pressure
Ar
Ar + air
Ar + H_2
Temperature range: 350–700°C
Digital multimeter with high impedance >1GW
Sensors were tested without metallic sheath (Mo electrode as auxiliary electric lead), so as to minimize unintentional contamination of the LBE with metals.



Sensor accuracy required for efficient oxygen control in HLMs

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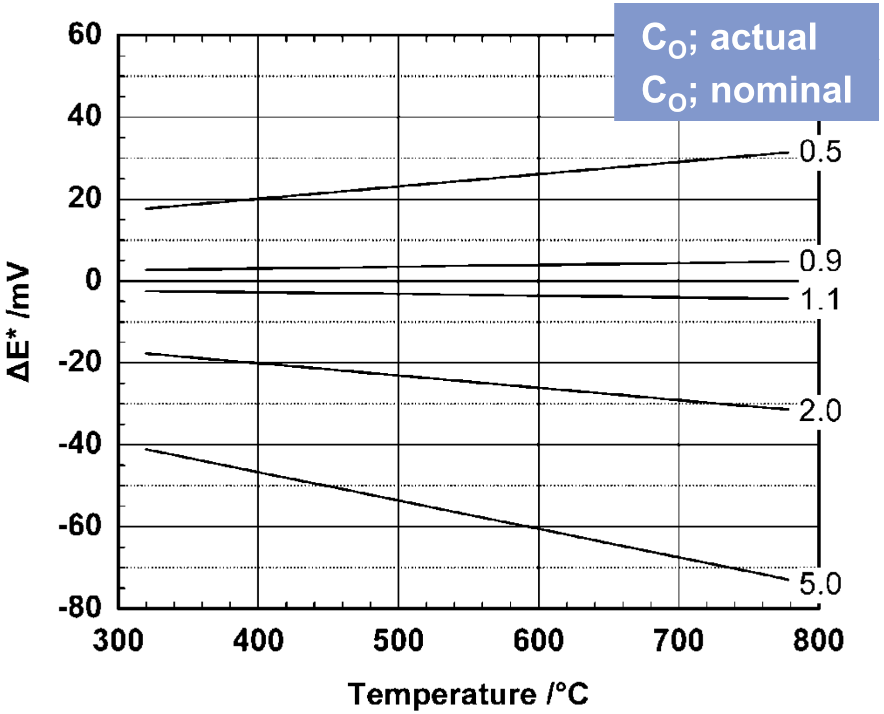


Experience

- Half an order of magnitude in oxygen concentration can significantly change oxidation mechanisms for F/M steels
- Reproducibility under service conditions better than +20 mV/-45 mV at 400°C and +30 mV/ -65 mV at 700°C is needed

Minimum requirement

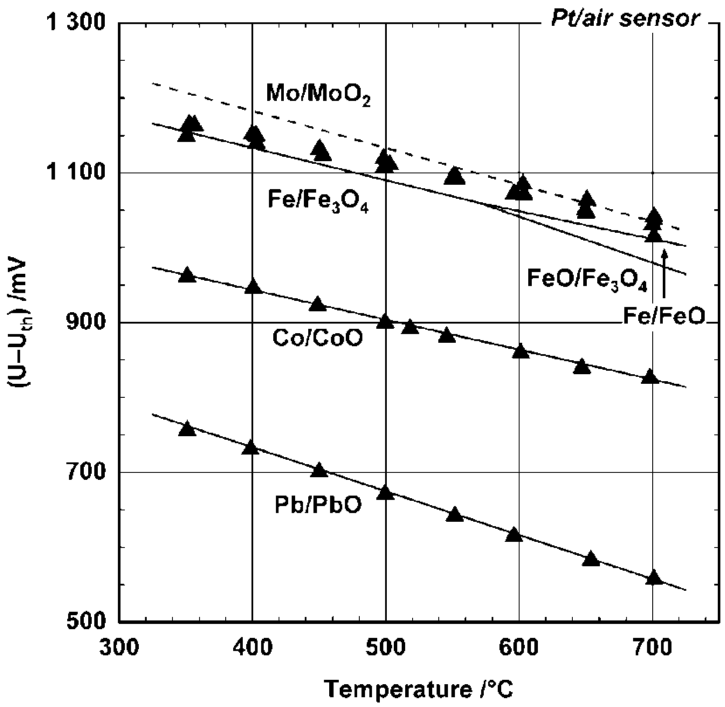
- Better than ± 20 mV at 400°C; ± 30 mV at 700°C
- Range of actual c_O from 0.5 to 2 c_O;nominal

Practical limit

- ± 5 mV, corresponding to ± 10% in c_O, resulting from uncertainty in thermodynamic data used for calculating reference potentials

Pt/air sensor and voltmeter with R_V > 1GΩ

Accuracy of measurement resulting from comparison with metal/metal-oxide equilibria adjusted in LBE



Fe oxide equilibria

- Stepwise cooling or heating
- Ar-15% H_2 bubbling continuously through the LBE (5 ml/min) or quasi-stagnant
- Oxygen potentials move from Fe-oxide to Mo/MoO_2 equilibrium with temperature variation (Mo comes from wire submerged in the LBE)

Co/CoO

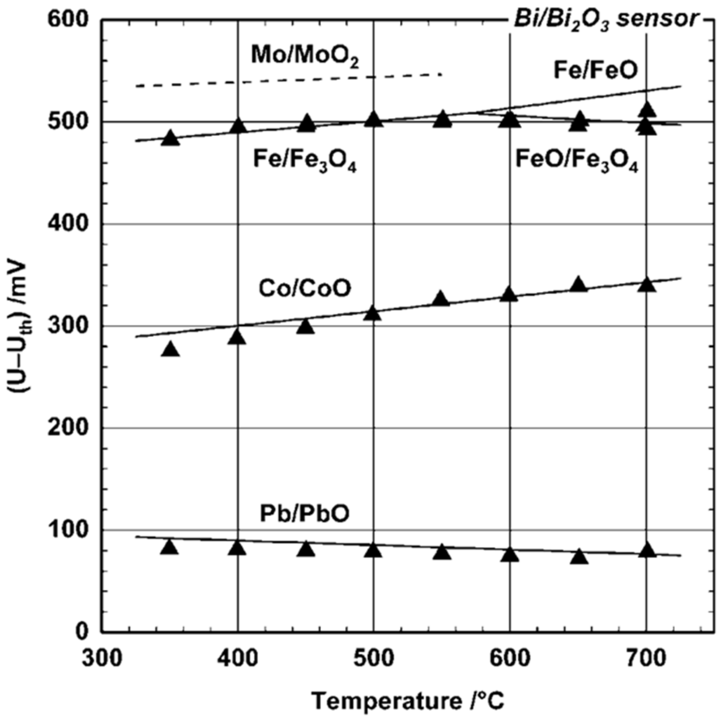
- Stepwise cooling
- Ar 5.0 bubbling continuously through the LBE (5 ml/min)
- Periodically addition of air (5 ml/min) at 700 and 650°C
- Maximum deviation from theoretical prediction < 6 mV

Pb/PbO

- Stepwise cooling
- Ar 5.0 bubbling continuously through the LBE (5 ml/min)
- Maximum deviation from theoretical prediction < 4 mV

Bi/Bi_2O_3 sensor and voltmeter with R_V > 1GΩ

Accuracy of measurement resulting from comparison with metal/metal-oxide equilibria adjusted in LBE



Fe oxide equilibria

- Stepwise cooling or heating
- Ar-15% H_2 mostly quasi-stagnant
- Maximum deviation from theoretical prediction < 8 mV

Co/CoO

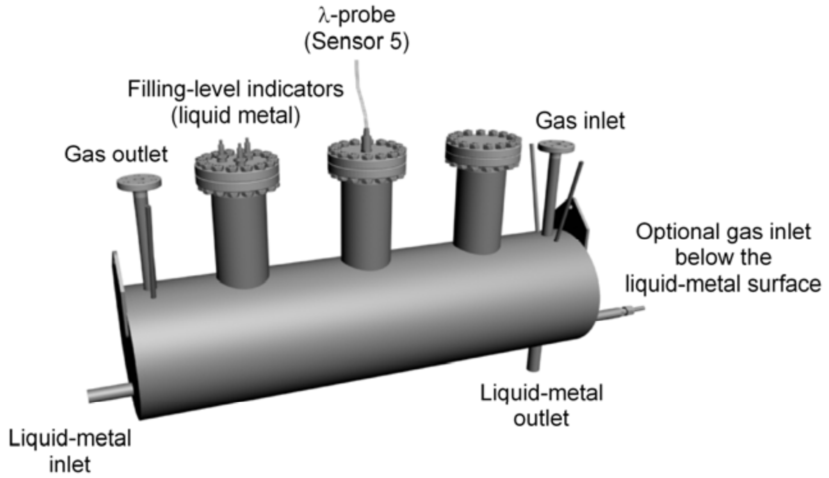
- Stepwise cooling
- Ar 5.0 bubbling continuously through the LBE (5 ml/min)
- Maximum deviation from theoretical prediction < 15 mV

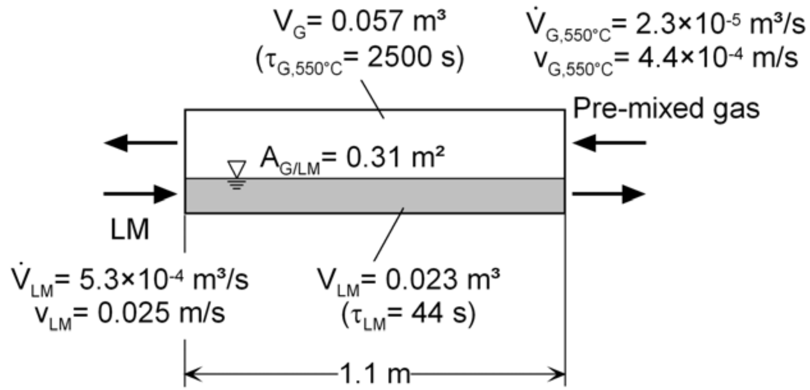
Pb/PbO

- Stepwise cooling
- Ar 5.0 bubbling continuously through the LBE (5 ml/min)
- Maximum deviation from theoretical prediction < 8 mV

Oxygen-transfer device of the CORRIDA loop

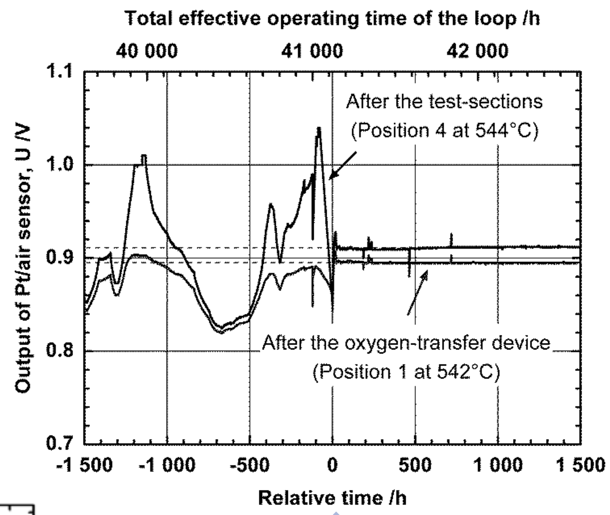
- **Gas/liquid**
  - Transfer across a plane liquid-metal surface
  - 5.3 kg/s LBE
  - ~500 cm³/min gas (referred to 25°C)
  - λ-probe for monitoring p_{O_2} in the gas-space
- **Gas mixture optimized for maintaining $c_O = 10^{-6}$ mass% at 550°C**
  - 500 cm³/min Ar humidified at 18°C (p_{H_2O} = 0.02 bar)
  - Continuous addition of 1–1.5 cm³/min ai (manually adjusted)
- **Gas mixture used for maintaining $c_O = 10^{-6}$ mass% at 450°C**
  - 500 cm³/min Ar humidified at 18°C (p_{H_2O} = 0.02 bar)
  - Discontinuous addition of 0.5 cm³/min air(manually adjusted)

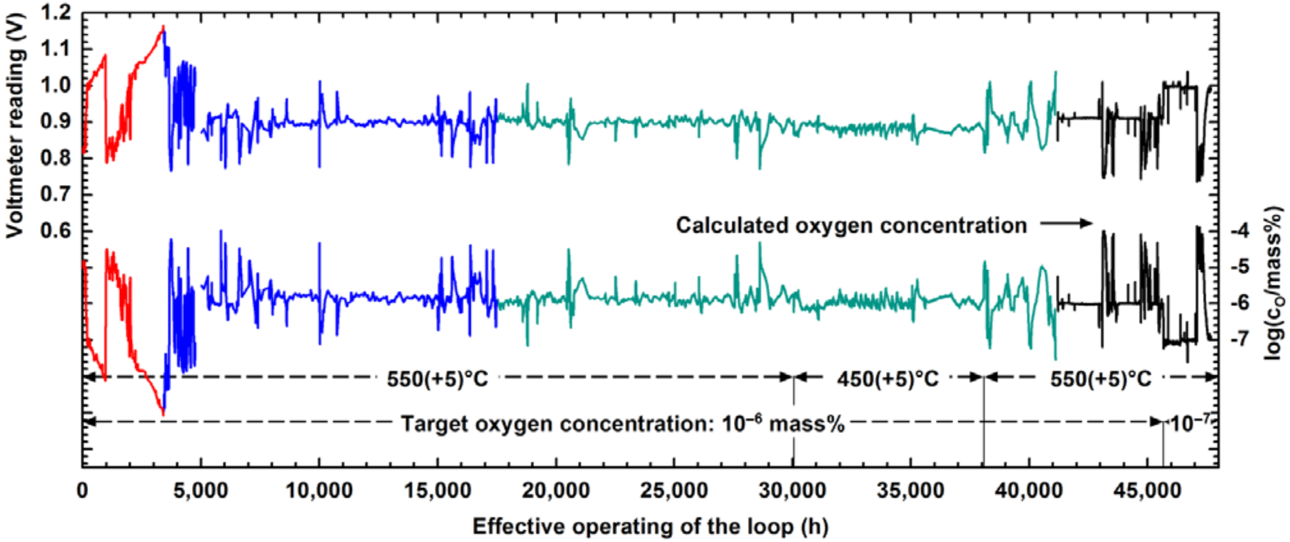




Performance of the oxygen-control system

- **Gas composition/control strategy**
  - Ar-H_2-H_2O corresponding to equilibrium at target oxygen concentration
  - Ar-H_2-H_2O, Ar-H_2O or Ar with varying air addition
  - Optimized manual air addition to Ar-H_2O
  - After implementation of automatic air addition to Ar or Ar-H_2





Before and after automatic air addition has been implemented

FM steels tested in the CORRIDA loop

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Concentration (in mass%) of alloying elements other than Fe

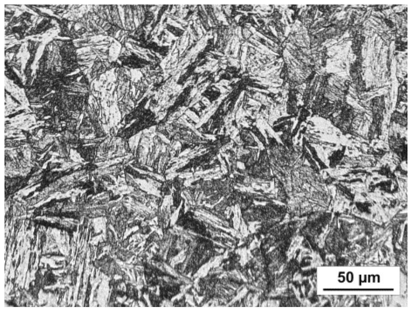
	Cr	Mo	W	V	Nb	Ta	Y	Mn	Ni	Si	C
T91-A	9.44	0.850	<0.003	0.196	0.072	n.a.	n.a.	0.588	0.100	0.272	0.075
T91-B	8.99	0.89	0.01	0.21	0.06	n.a.	n.a.	0.38	0.11	0.22	0.1025
E911*	8.50–9.50	0.90–1.10	0.90–1.10	0.18–0.25	0.060–0.100	–	–	0.30–0.60	0.10–0.40	0.10–0.50	0.09–0.13
EUROFER	8.82	<0.0010	1.09	0.20	n.a.	0.13	n.a.	0.47	0.020	0.040	0.11
EF-ODS-A	9.40	0.0040	1.10	0.185	n.a.	0.08	0.297†	0.418	0.0670	0.115	0.072
EF-ODS-B	8.92	0.0037	1.11	0.185	n.a.	0.078	0.192†	0.408	0.0544	0.111	0.067

* Nominal composition
† In the form of yttria (Y_2O_3)

Nominally 9 mass% Cr

⇧ ⇧ Elements besides Cr that are likely to improve oxidation performance

Microstructure

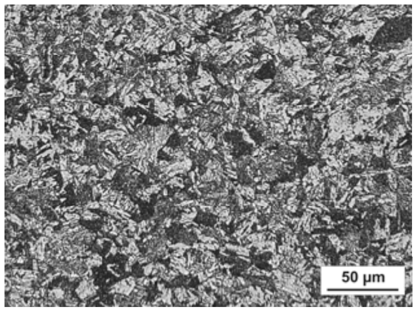


Fully martensitic: E911, T91-A

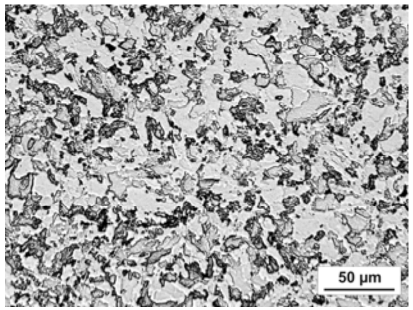
T91-B

Grain Size

EUROFER



Mainly ferritic: ODS-A, ODS-B



MYRRHA design data used

Design operating parameters

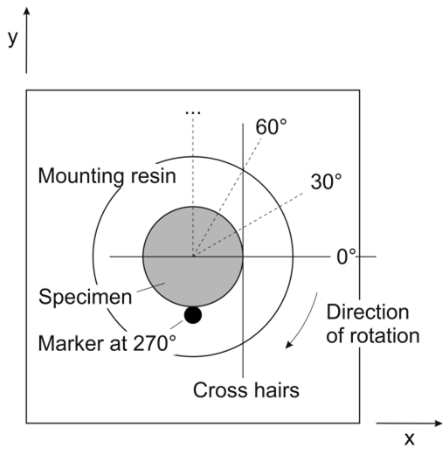
Material	Temperature (°C)	Surface area (m^2)	Oxygen conc. (mass%)	Average flow velocity (m/s)
316L	400	120	~10^{-7}	To be specified
	350	419 (360)		
	310	125		
	270	1697 (1596)		
15-15Ti	410	194	~10^{-7}	To be specified
	270	1150		
T91	450	4	~10^{-7}	To be specified
	350	(59)		
	270	1 (102)		

Early operating stages

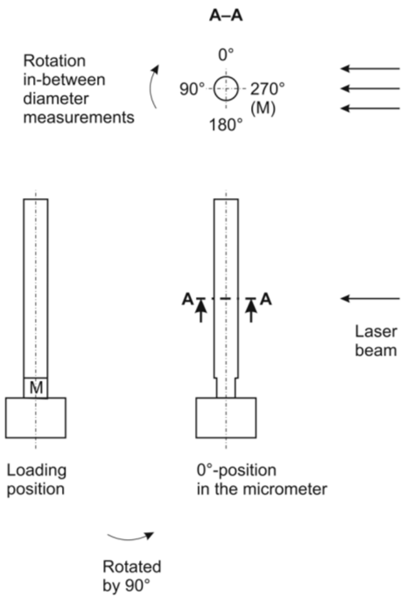
	Duration (days)	Temperature (°C)
Start-up	180	270°C
1st Power cycle	90	Components at design operating temperature; temperature transients
Downtime	90	270°C
2nd Power cycle	90	As above

Quantification of corrosion

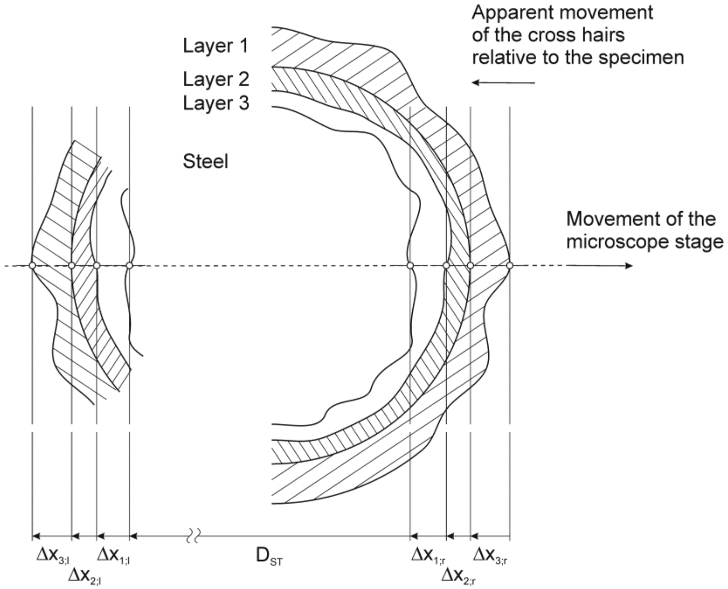
- **Goal of quantification**
  - Material loss, average of general corrosion and maximum of local corrosion
  - Thickness of adherent (oxide) scale
  - Overall change in dimensions, including the scale
  - Amount of metals transferred to the liquid metal



- **Metallographic method (cylindrical specimens)**
  - Measurement of initial diameter in a laser micrometer with 0.1 µm resolution



  - Diameter of unaffected material and thickness of corrosion scales determined in a microscope (LOM) at minimum ×500 magnification, with 1 µm resolution
  - Occurrence of different corrosion modes on opposing sides of the re-measured diameter is considered in the evaluation

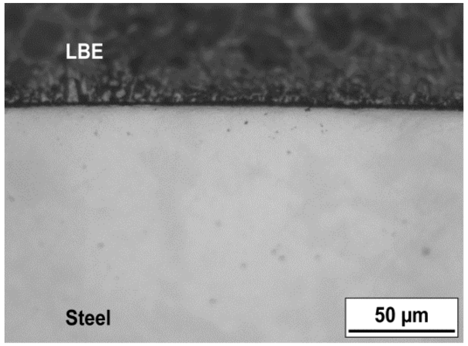


Detailed quantification procedure

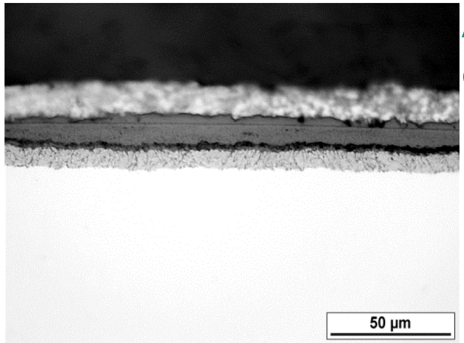
- **On the evaluated cross section**
  - Start at 0° position
  - Identify corrosion mode in this position
  - Measure scale thickness
  - Measure diameter sound material
  - Identify corrosion mode and quantify scale on the other side of the diameter (180°)

- **Respective measurements after turning by 30°**
  - Overall 12 sets of measurements
  - 6 independent (0°, 30°, 60° …)
  - Each one repetition (180°, 210°, 240° …)

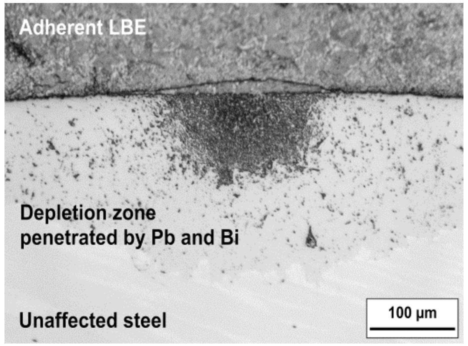
→ **First round of measurements (partners quantify their own experiments) almost finished.**
Second round will be started as soon as possible.



Protective scaling

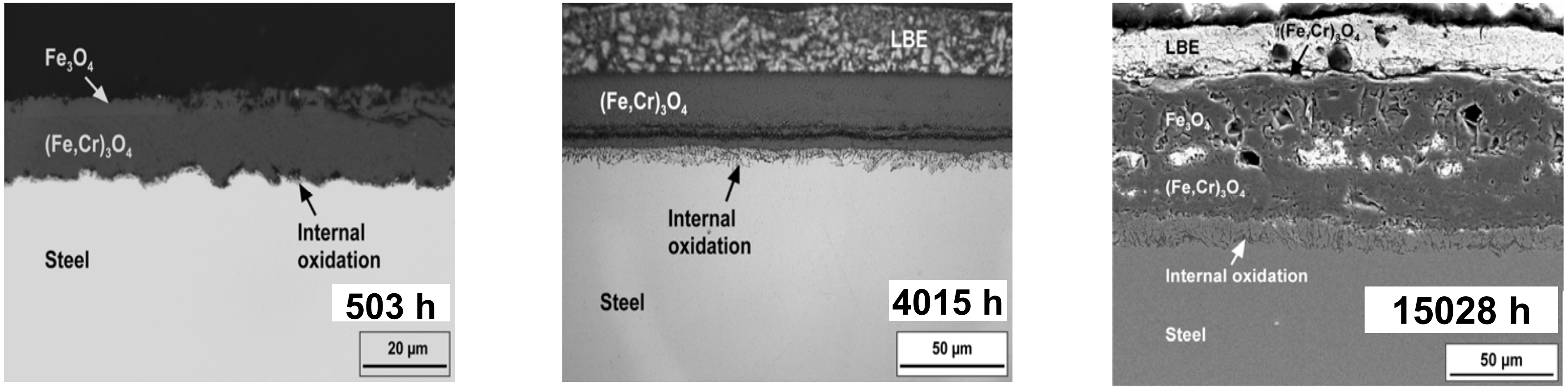


Accelerated oxidation



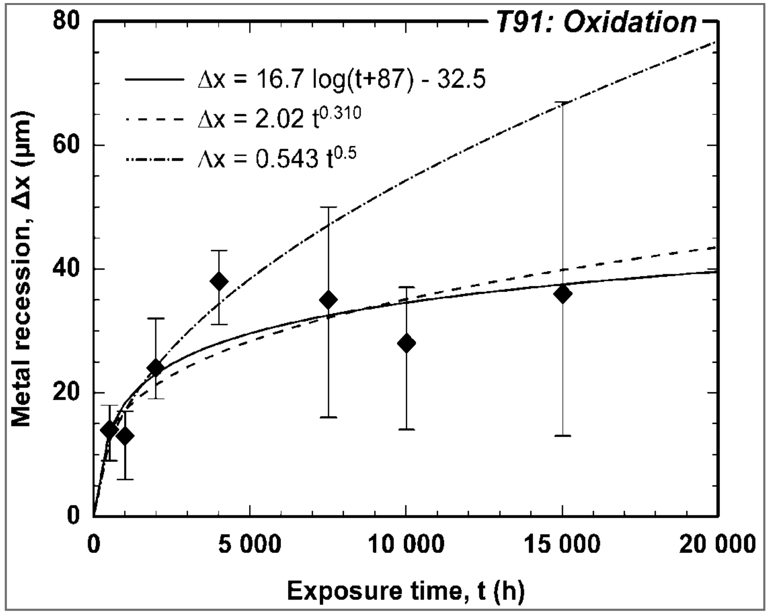
Solution-based corrosion (selective leaching)

T91: Qualitative performance in oxygen-containing LBE at 550°C, v = 2 m/s and $c_O = 1.6\times10^{-6}$ mass% (I)



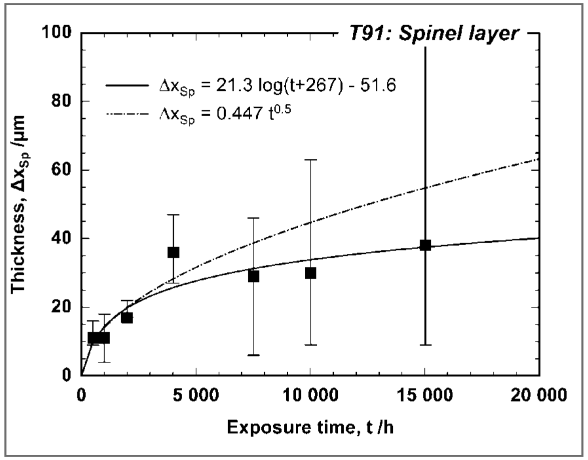
- Oxidation
- Oxide scale consists of
  - Magnetite (Fe_3O_4)
  - Cr-deficient spinel ($Fe(Fe_xCr_{1-x})_2O_4$)
  - Internal Oxidation Zone (IOZ)
- Magnetite is mostly missing, i. e., Fe is partially dissolved by the liquid metal (or eroded after Fe_3O_4 formation?)
- Inclusions of Pb and Bi inside the scale, espesially after long exposure times

T91: Quantification of oxidation in oxygen-containing LBE at 550°C, v = 2 m/s and $c_O = 1.6\times10^{-6}$ mass% (II)



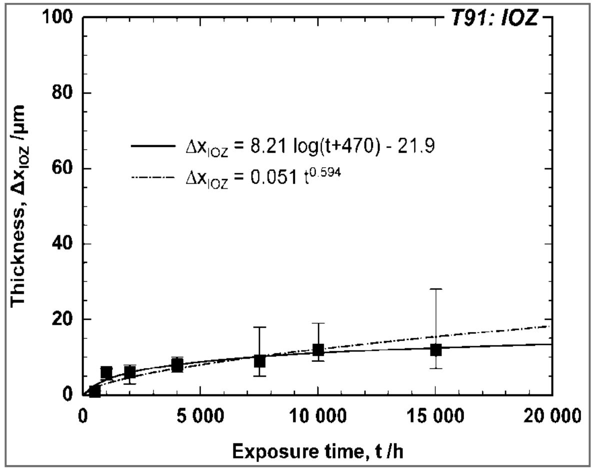
- Metal recession (loss of cross-section)
- Compromises the structural integrity of plant components
- Determined from measurements in the LOM (generally six measurements per investigated cross-section)
- Includes internal oxidation
- Local variation significantly increases with increasing exposure time
- Optimistic prediction: 50–70 µm after 100,000 h
- Worst-case: 100 µm after 4 years

T91: Quantification of oxidation in oxygen-containing LBE at 550°C, v = 2 m/s and $c_O = 1.6 \times 10^{-6}$ mass% (III)

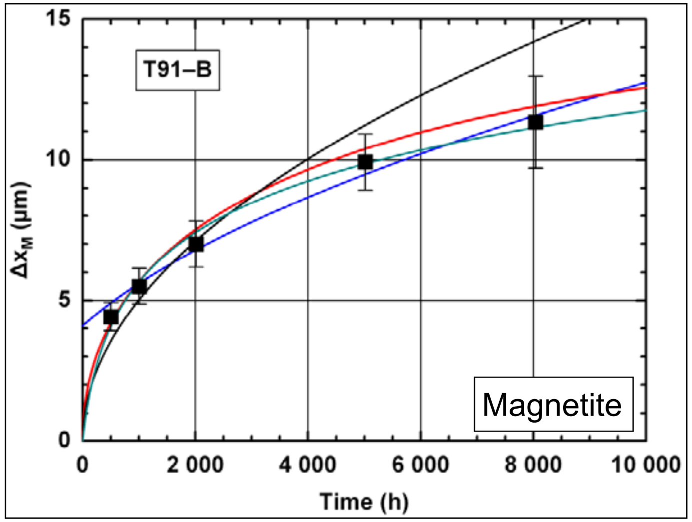


- Thickness of different layers of the oxide scale
- May affect heat transfer in the case of thermally-loaded plant components
- Generally twelve measurements per investigated cross-section
- Thickness of spinel layer significantly varies locally with increasing exposure time
- Average thickness of the spinel layer is in the order of the metal recession

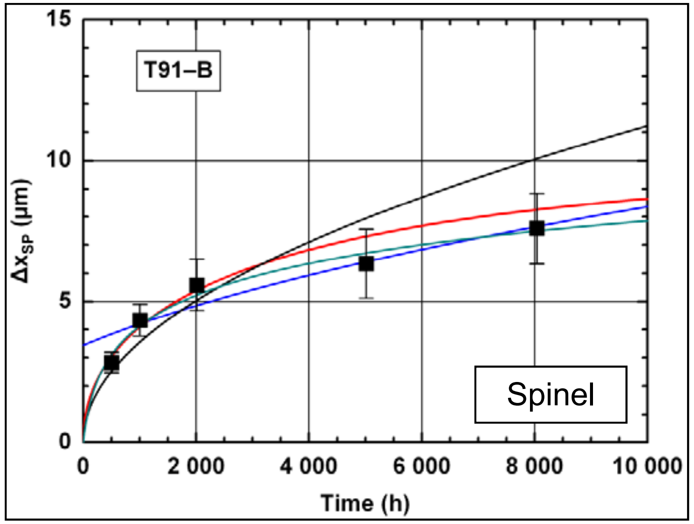
Fe flux into the LBE can be estimated from the spinel layer thickness



Kinetics of oxide-scale growth for T91-B at 450°C, 2 m/s and 10^{-6} mass% oxygen (I)

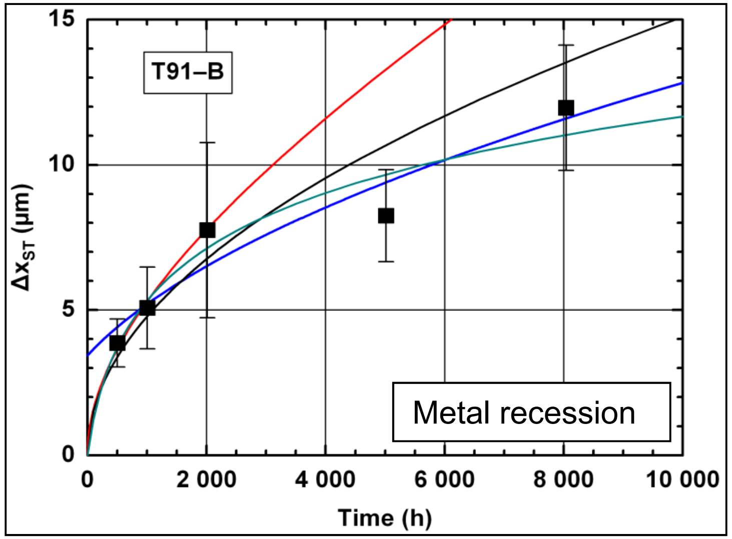


Parabolic:	$\Delta x^2 = k_2 t$
Parabolic after faster initial kinetics:	$\Delta x^2 = k_2 t + C_2$
Logarithmic:	$\Delta x = k_{log} \log(t + t_0) + C_{log}$
Paralinear:	$\frac{d\Delta x}{dt} = \frac{k_p}{d\Delta x} + k_1$



- **Local internal oxidation was not considered**
- Thickness of the oxide layers slightly lower (by ~20%) for T91-A

Data extrapolation for T91 at 450°C, 2 m/s and 10^{-6} mass% oxygen (II)



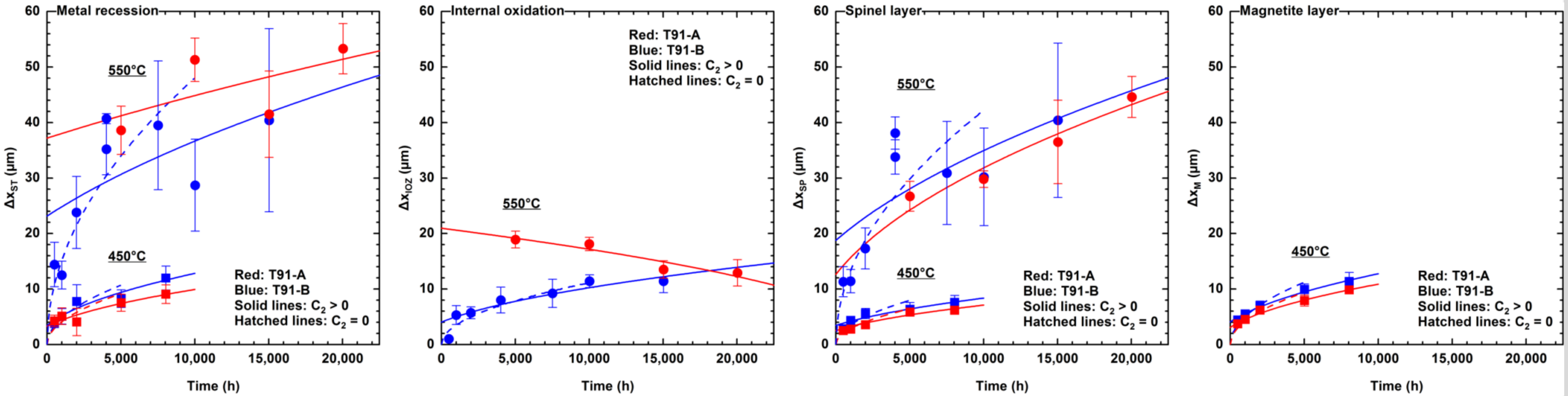
Exposure time (years)	1	5	10
T91-A → Upper limit of Cr content specified for T9			
Δx_M (μm)	10	13 – 22	13 – 31
Δx_{SP} (μm)	7	8 – 14	8 – 20
Δx_{ST} (μm)	9	20	28
T91-B → Lower limit of Cr content specified for T91			
Δx_M (μm)	12	15 – 26	15 – 36
Δx_{SP} (μm)	8	10 – 16	10 – 23
Δx_{ST} (μm)	12	26	37

Parabolic:	$\Delta x^2 = k_2 t$
Parabolic after faster kinetics:	$\Delta x^2 = k_2 t + C_2$
Paralinear model of oxide scale growth	
Logarithmic:	$\Delta x = k_{log} (t + t_0) + C_{log}$

Quantitative results from experiments in the CORRIDA loop: T91 in LBE at 450–550°C, 2 m/s and 10^{-6}% dissolved oxygen

Karlsruhe Institute of Technology

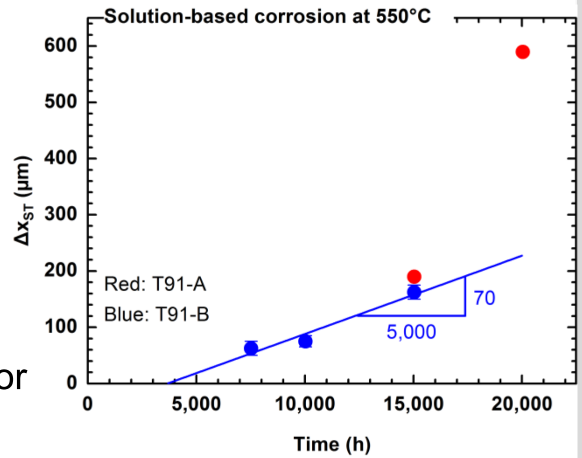
❑ Accelerated oxidation



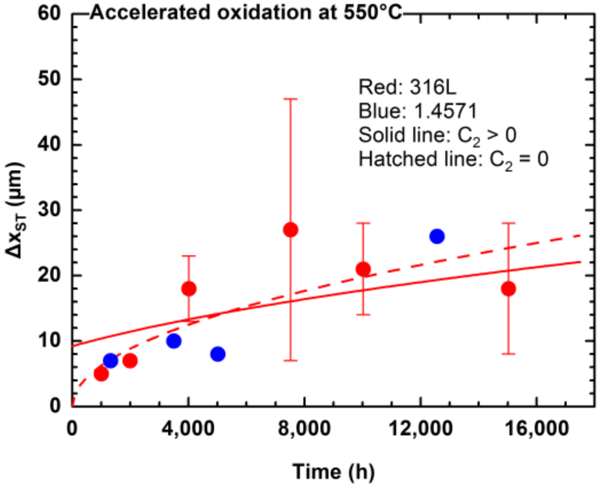
- Thickness of magnetite approximates the overall increase in specimen radius
- Dissolved Fe from balancing the mass consumed and present in oxides
- Extrapolation of data naturally depends strongly on the type of rate law assumed

❑ Solution-based corrosion

- Significantly increased material loss
- Comparatively small database for kinetic analysis
- Underlying corrosion mechanisms may differ for the particular data points

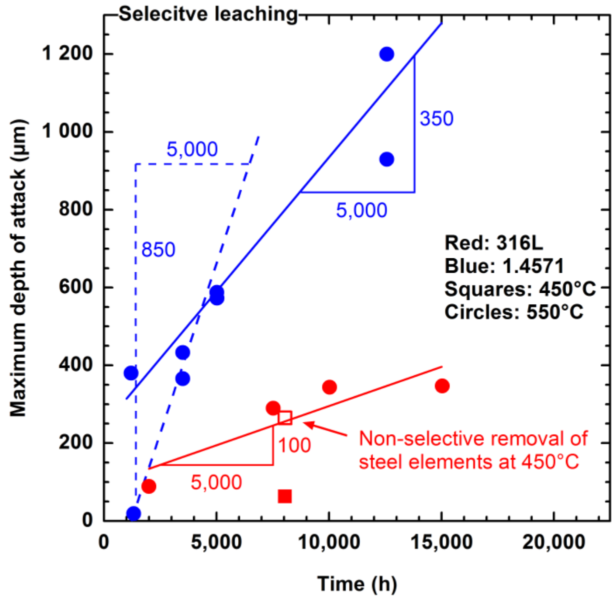


Quantitative results from experiments in the CORRIDA loop: Type 316 in LBE at 450–550°C, 2 m/s and 10^{-6}% dissolved oxygen



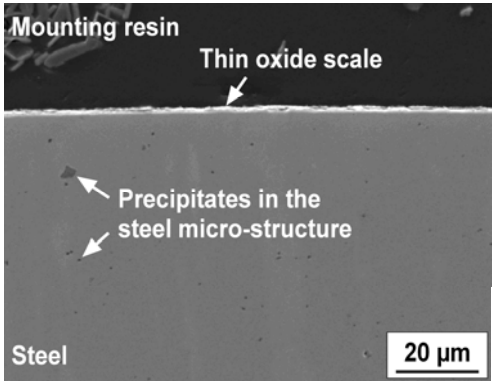
- **Accelerated oxidation**
  - Observed locally at 550°C
  - In parts continuous scale after long exposure time
  - Not observed at 450°C

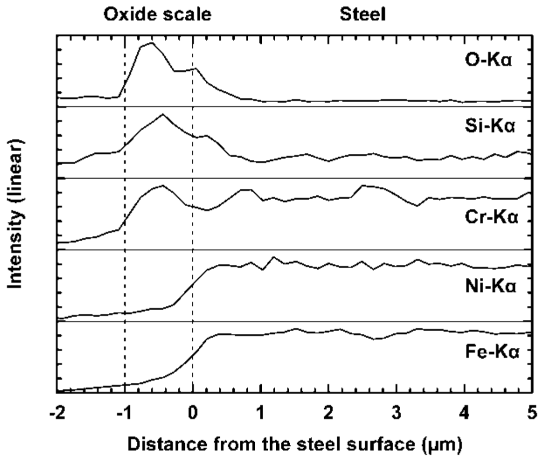
- **Solution-based corrosion**
  - Only few sites on investigated specimens may be affected
  - Mostly selective leaching of Ni and Cr
  - But also general dissolution of all steel elements at 450°C
  - Incubation time decreases from around 5000 h at 450°C to 1000 h at 550°C



Corrosion of type 316-Ti austenitic steels in oxygen-containing LBE (1)

- **Protective scaling**
  - Thin oxide scale (< 1 µm) consisting of Cr- or Si-rich oxide layers
  - Might have evolved from thin films already existing on the steel surface before exposure
  - Similar to the scale formed by pre-oxidation in dry gas (Ar)
  - Locally long-lasting phenomenon on specimens exposed at 450/550°C, 10^{-6} mass% O in the test-sections of the loop
  - Not observed on tube samples taken from the hot leg of the loop; effect of long exposure time and variiying c_O (?)



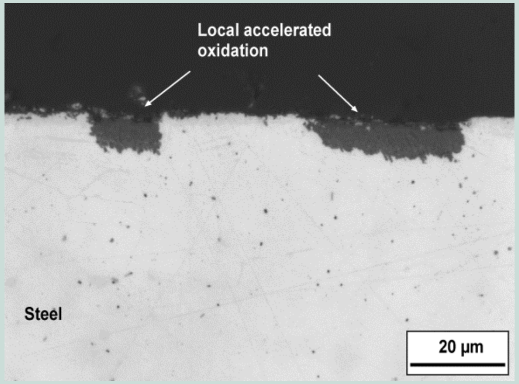


SS 316-Ti specimen in the test-section of the loop after exposure for 3495 h to oxygen-containing flowing LBE at 550°C and cO ≈ 10–6 mass%

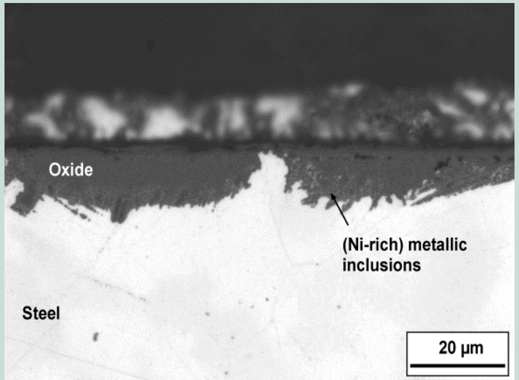
Corrosion of type 316-Ti austenitic steels in oxygen-containing LBE (2)

- **Accelerated oxidation**
  - Starts locally where the thin oxide scale lost integrity or did not form
  - Formation of $Fe(Fe_xCr_{1-x})_2O_4$, Fe_3O_4 and an internal oxidation zone; the latter two depending on oxygen content, temperature (or flow velocity)
  - The thicker scale spreads on the steel surface with time and becomes partially continuous
  - Varying c_O (mostly lower than 10^{-6} mass%) seems to promote accelerated oxidation

SS 316-Ti specimens in the test-sections of the loop:

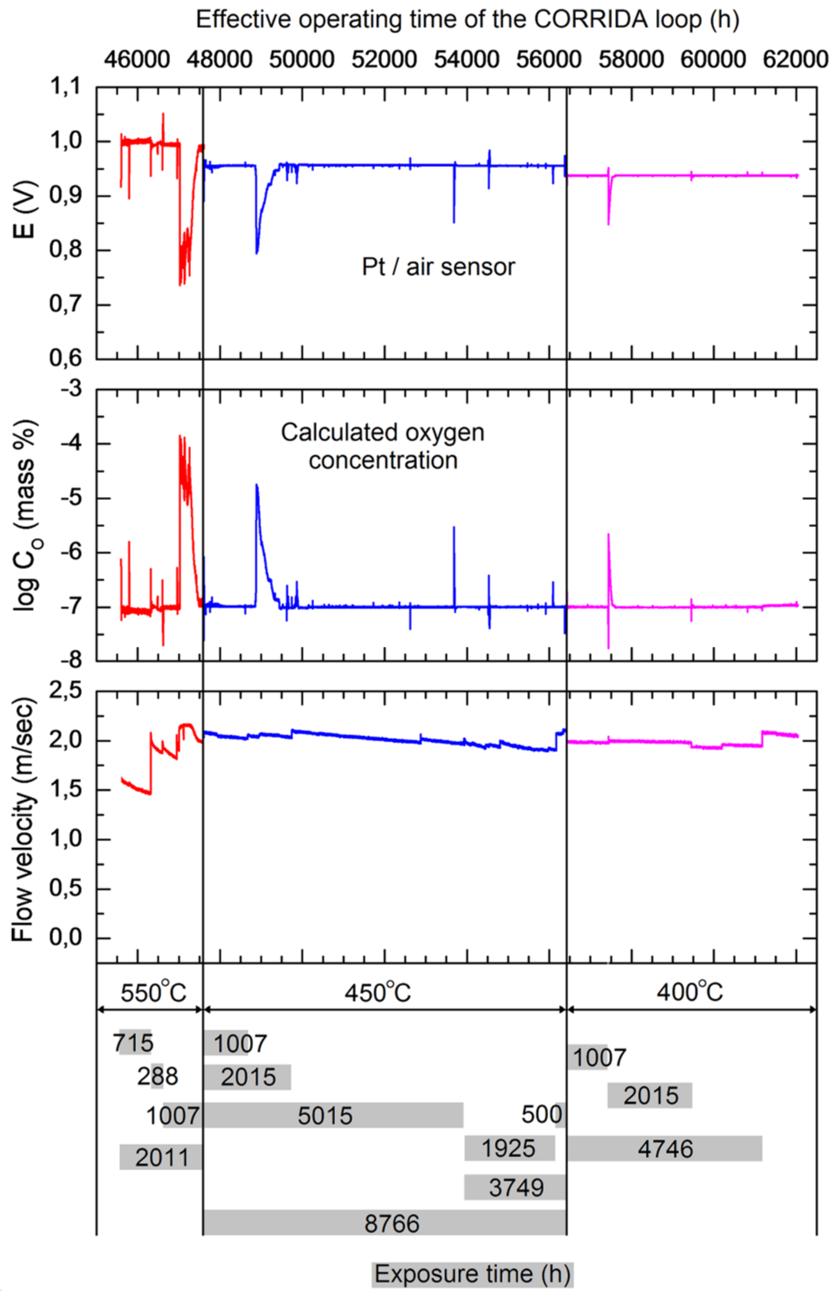


After exposure for 3495 h at 550°C and $c_O \approx 10^{-6}$ mass%



After exposure for 10,006 h at 550°C and variying c_O

Experiments in CORRIDA



Materials	316 L (plate), 1.4571 (rod), 1.4970 (rod), T91 (two different plates)
Liquid metal	LBE

Temp. /°C	550	450	400
Oxygen /mass%	10^{-7}*	10^{-7}†	10^{-7}
Flow velocity /(m s^{-1})	2‡	2	
T_{min} /°C (Loop)	~385	~350	~350
Exposure time /h	~300–2000 (4 samples)	~500–9000 (7 samples)	~1000–5000 (3 samples)

* Temporary excursion to ~10^{-4} mass% for 450 h, after 1450 h total runtime of the experiment.

† Temporary excursion to ~10^{-5} mass% for 600 h, after 1200 h total runtime of the experiment.

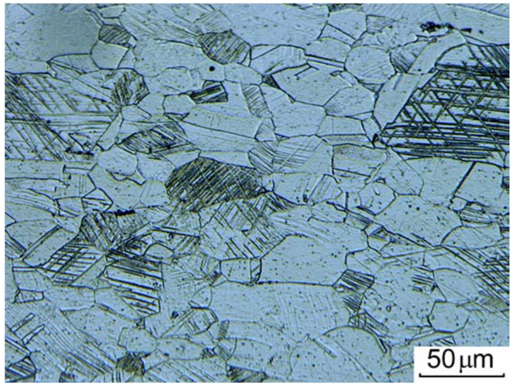
‡ Varying flow velocity, around 1.5 m/s during the first 700 h of total runtime of the experiment.

1.1 Materials

(Fe – Bal.)

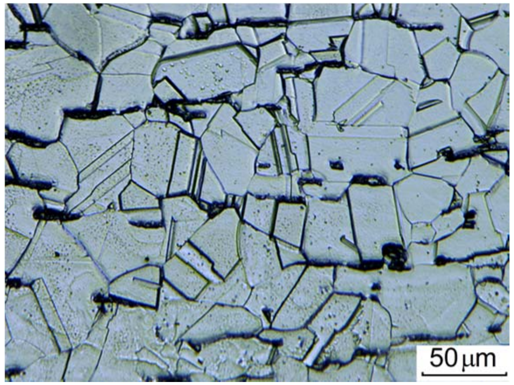
Austenitic steels	Cr	Ni	Mo	Mn	Si	Cu	V	W	Al	Ti	C	N	P	S	B
316L	16.73	9.97	2.05	1.81	0.67	0.23	0.07	0.02	0.018	-	0.019	0.029	0.032	0.0035	-
1.4970	15.95	15.4	1.2	1.49	0.52	0.026	0.036	< 0.005	0.023	0.44	0.1	0.009	< 0.01	0.0036	< 0.01
1.4571	17.50	12	2.0	2.0	1.0	-	-	-	-	0.70	0.08	-	0.045	0.015	-

1.4970 (15-15Ti)



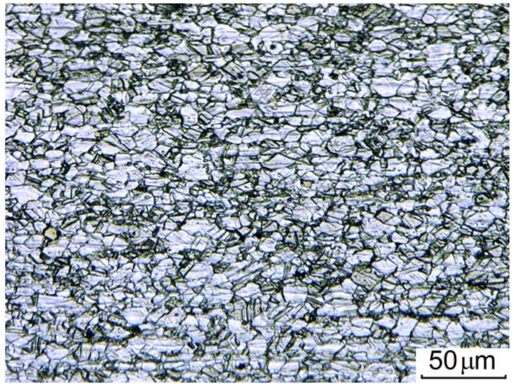
- HV_{30} = 253;
- Grain size ranged from 20 to 65 µm;
- Intersecting deformation twins.

316L



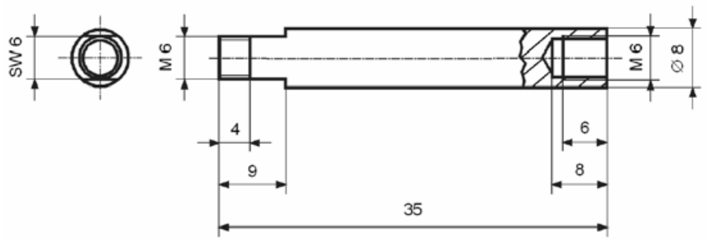
- HV_{30} = 132;
- Grain size averaged 50 µm (G 5.5);
- Annealing twins.

1.4571

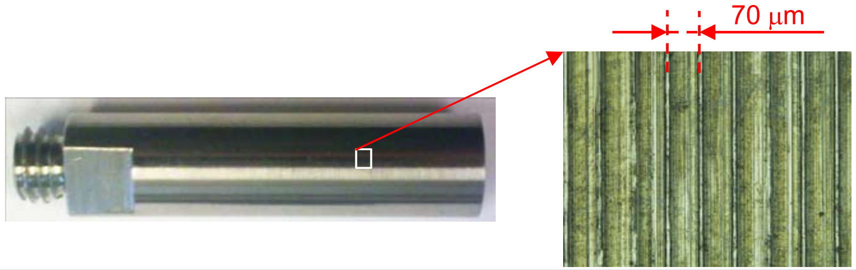


- HV_{30} = 245;
- Grain size averaged 15 µm (G 9.5).

Shape and dimensions of sample for corrosion tests

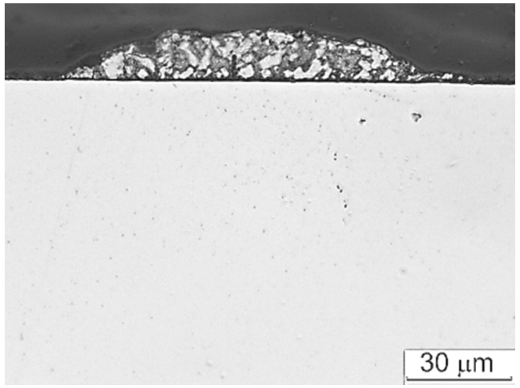


General view of initial sample after finishing turning



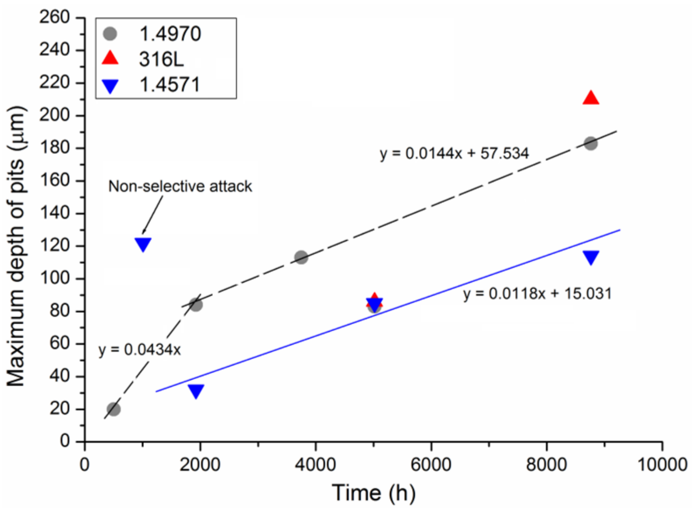
Austenitic steels after exposure in the CORRIDA loop

- **400°C/ 10^{-7} mass% oxygen**
  - Only protective scaling at up to 4766 h

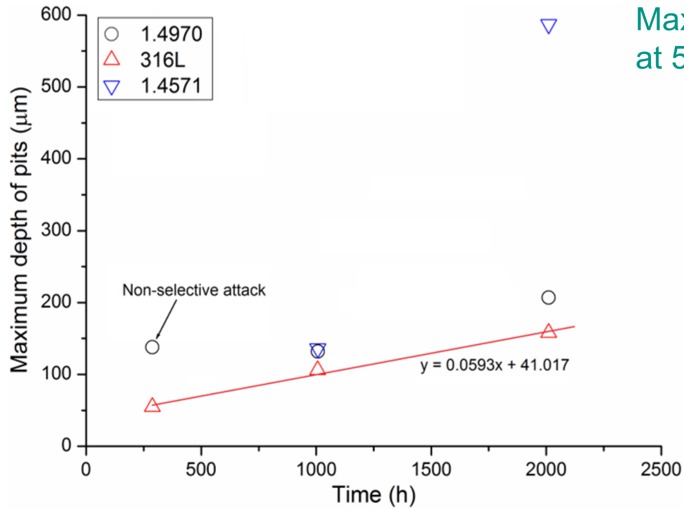


1.4970
after 4766 h
at 400°C/10^{-7}%

- **450 and 550°C/ 10^{-7} mass% oxygen**
  - Protective scaling and selective leaching (SL)
  - Occasionally non-selective attack (general solution) at higher rate than SL
  - Comparatively long incubation of SL seems coupled to faster progress
  - Steel that showed highest relative resistance against SL at 450°C, corrodes fastest at 550°C (1.4571), and vice versa (316L)
  - 100–200 µm local material loss after 9000 h at 450°C, 150–600 µm after 2000 h at 550°C



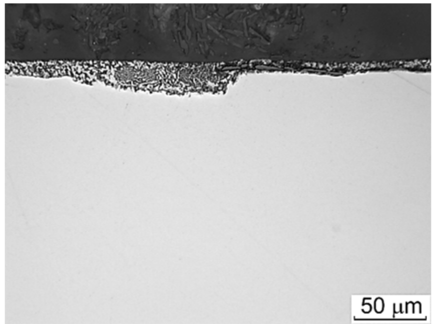
Maximum of SL
at 450°C/10^{-7}%



Maximum of SL
at 550°C/10^{-7}%

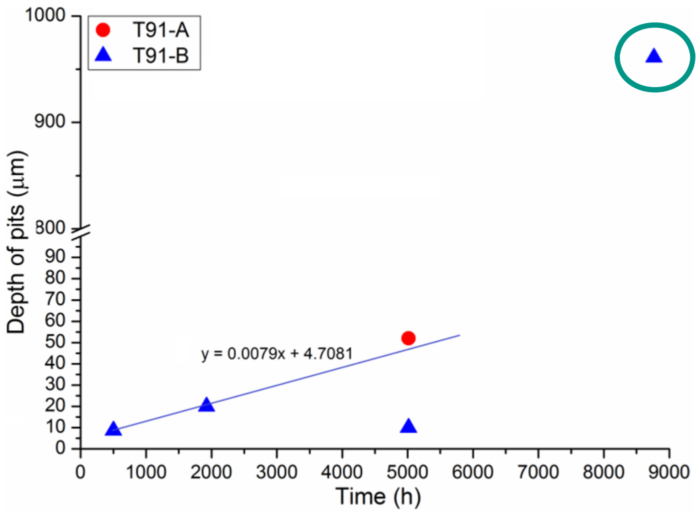
T91 after exposure in the CORRIDA loop

- **400°C/ 10^{-7} mass% oxygen**
  - Primarily accelerated oxidation (AO)
  - Flawed and partially detached oxide scale
  - Solution-based corrosion (SB) observed locally after 4766 h

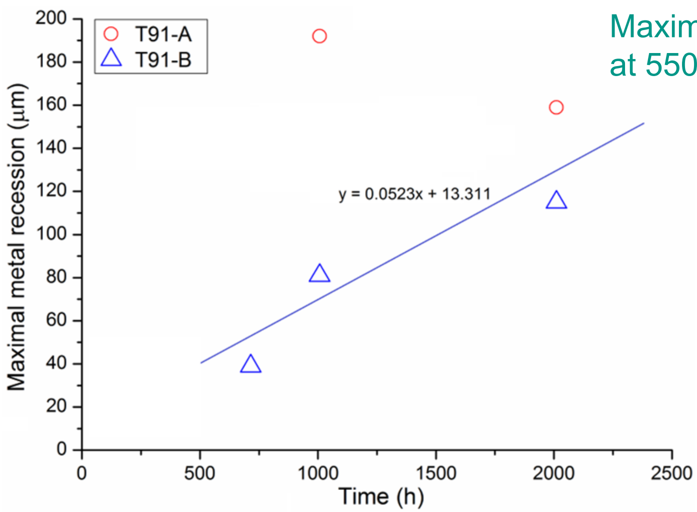


T91-B after 4766 h
at 400°C/10^{-7}%

- **450 and 550°C/ 10^{-7} mass% oxygen**
  - Protective scaling locally still evident, especially after shorter exposure time
  - Dominant AO
  - Possible incipient stages of SB after 500, clearly observed after 5000 h at 450°C
  - At 550°C, incubation of SB between ~300 and 700 h
  - ~50 µm maximum SB after 5000 h at 450°C, exceptionally severe attack observed on T91-B (950 µm) after 8766 h
  - Maximum 190 µm after 1000 h at 550°C



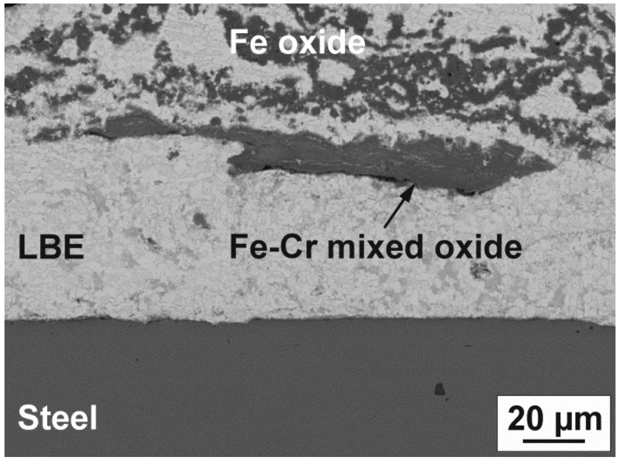
Maximum of SB
at 450°C/10^{-7}%



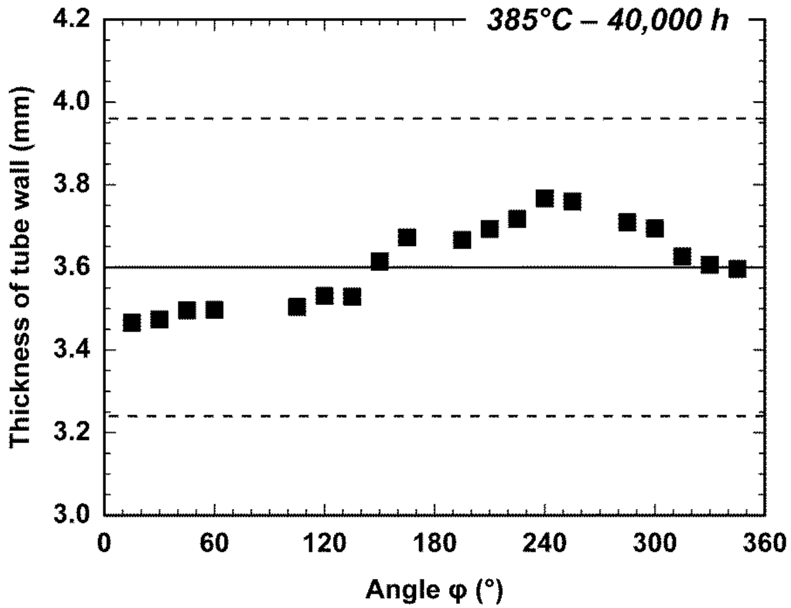
Maximum of SB
at 550°C/10^{-7}%

Performance of the tubing of the CORRIDA loop

- **Sample T5 after 40,000 h at 385°C**
  - Position after the cooler, before magnetic trap
  - No significant change in wall thickness after the long-term exposure
  - Oxide deposits in adherent solidified LBE, but only in some distance from the tube wall
  - Neither deposits nor significant amounts of oxide on the surface



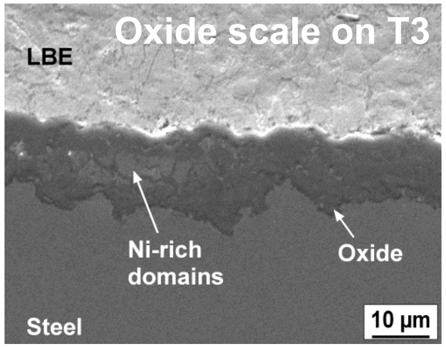
Electron-optical micrograph (BSE)

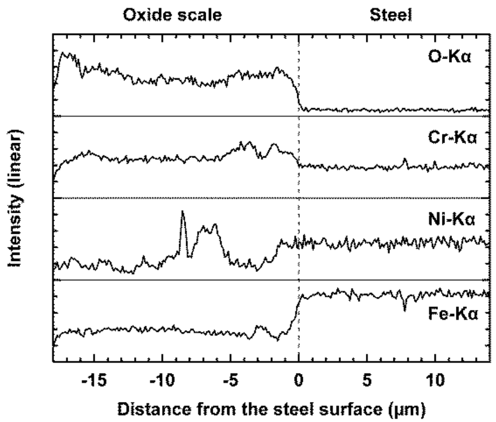


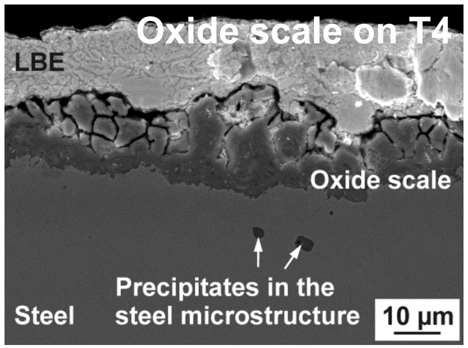
Results from measuring the residual wall thickness in the microscope

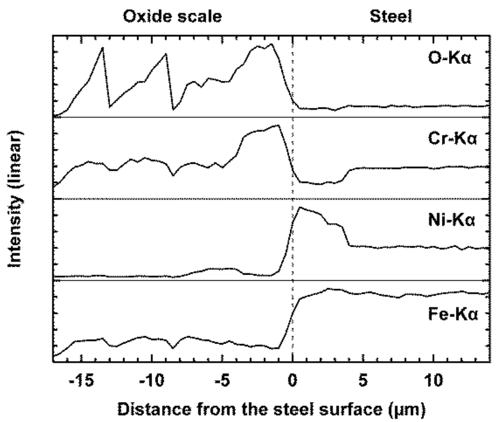
Performance of the tubing of the CORRIDA loop

- **Corrosion scales formed in the hot leg (550°C)**
  - T3 (6000 h) and T4 (40,000 h) mainly show oxidation; T3 was not pre-oxidised
  - T1 (23,000 h) and T2 (29,000 h) show significant selective leaching

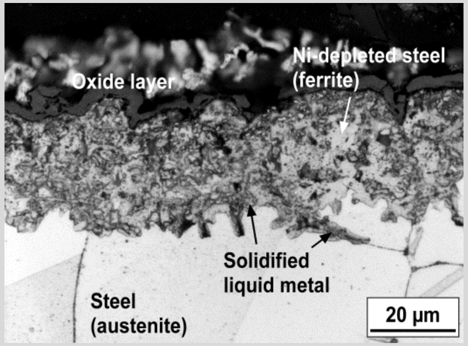


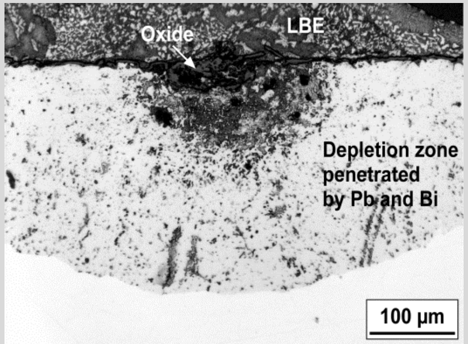






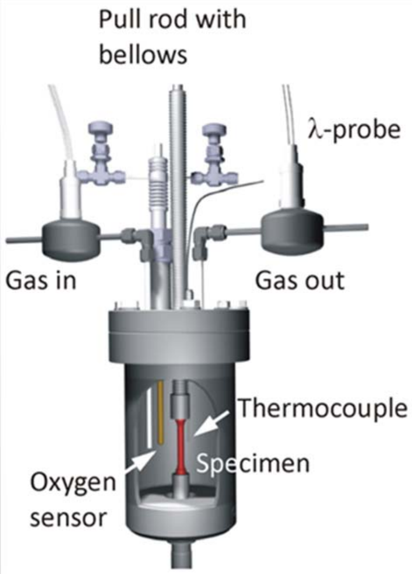
Corrosion scales typically observed on T1 and T2





Creep-to-Rupture tests in stagnant, oxygen-controlled liquid Pb at 650°C

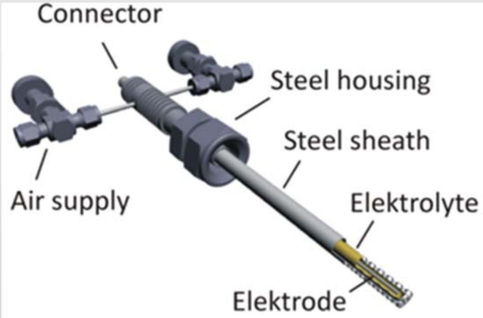
CRISLA-capsule



Facility with infrastructure:

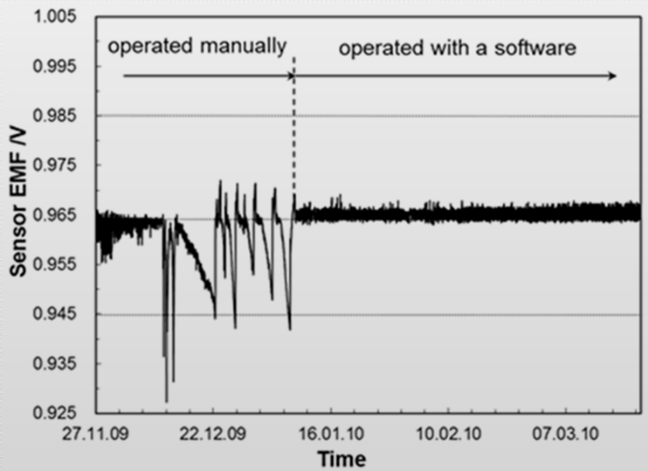
- 5 capsules for stagnant Pb with volume of 900 ml. Oxygen control periphery for each capsule.
- 3 capsules for stagnant air with volume of 230 ml

Pt/air oxygen sensor



Gas supply:

- Ar (continuous) – 96-99 ml/min
- Ar/H_2 (continuous) – 3 ml/min
- synthetic air (pulsed) – 1ml/min if E≥965 mV



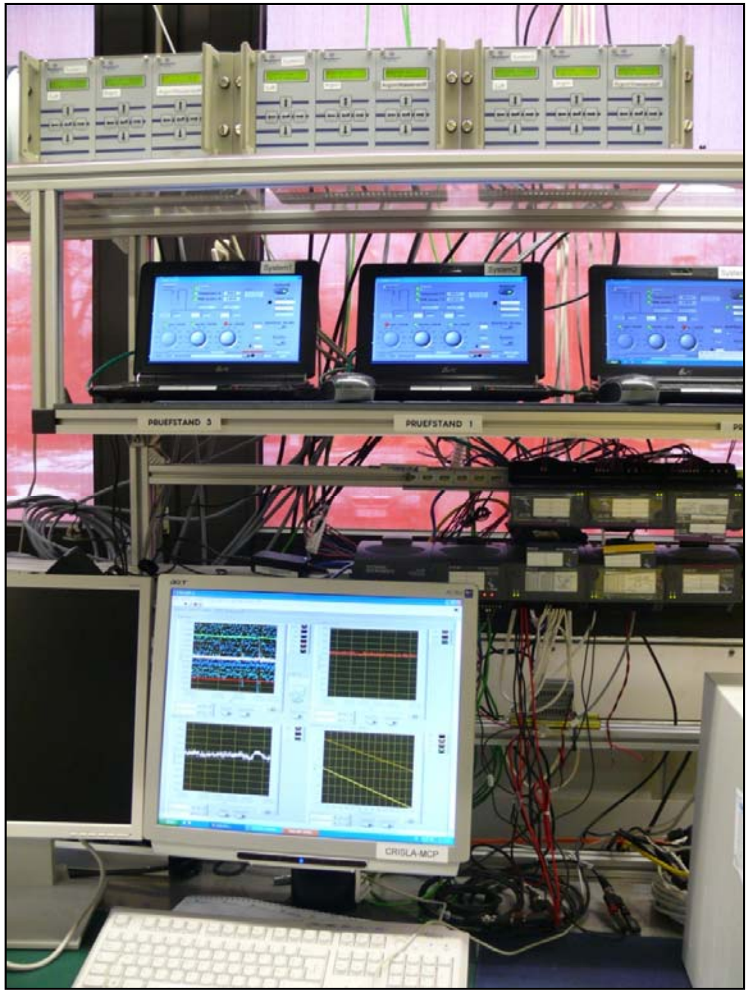
- stagnant Pb or LBE
- T = 450–650°C
- $c_O = 10^{-7}–10^{-6}$ mass.-%

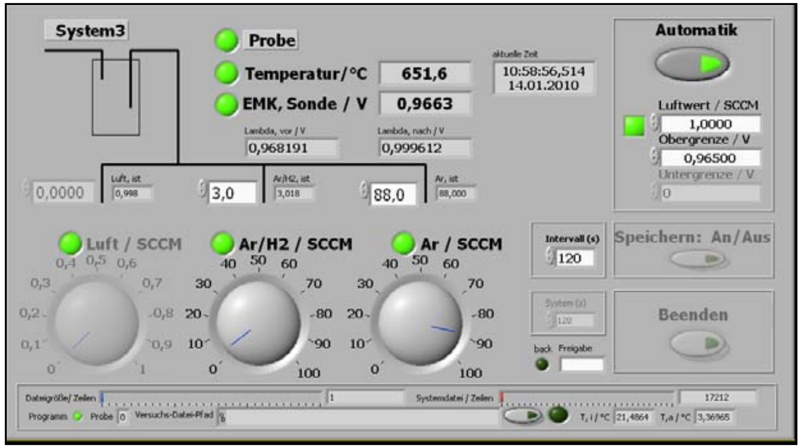
- through oxygen contained gas (gas/liquid oxygen-transfer)

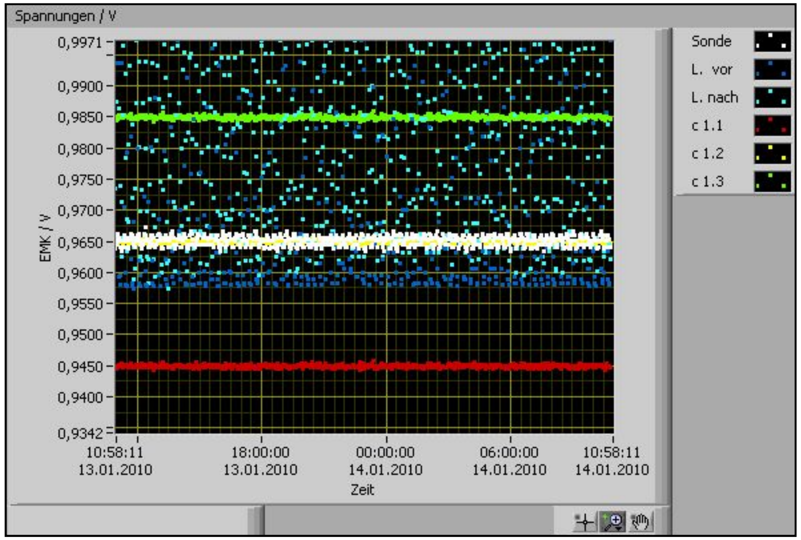
- E: 965±20 mV → 965±2 mV

CRISLA Facility for Creep-Rupture Tests in Lead

PC-supported control system for oxygen content: user defined settings

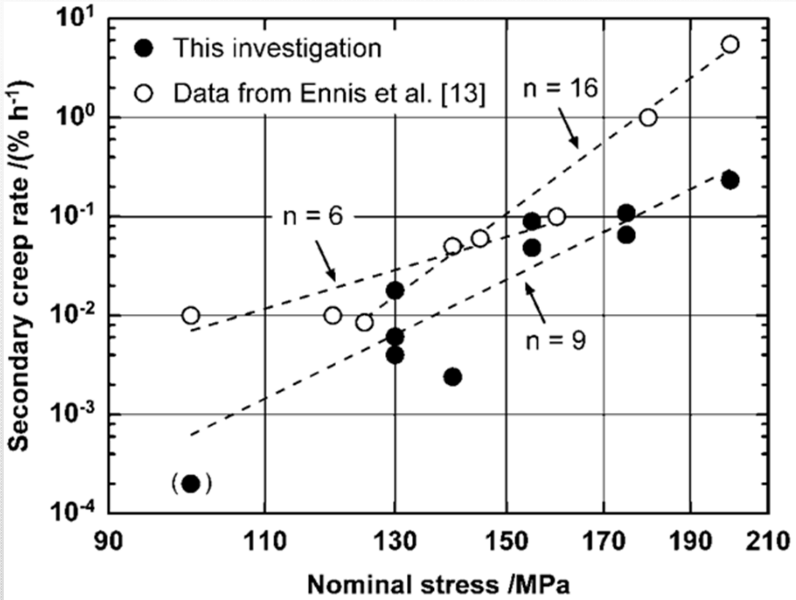


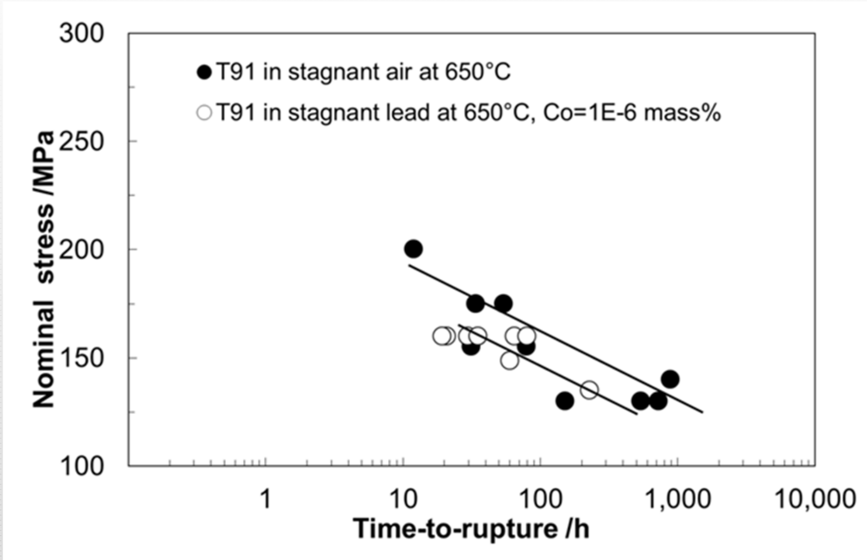




Creep strength of T91 in air and lead at 650°C

Experimental and literature data for T91 in air



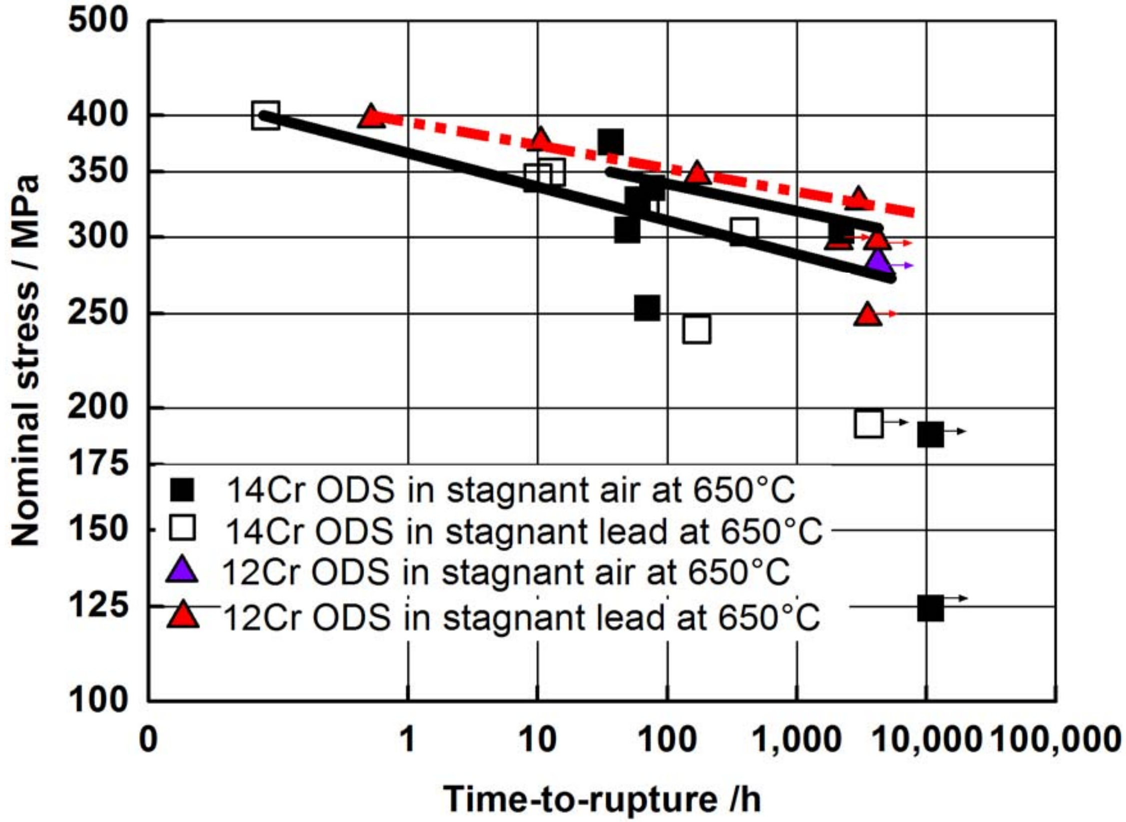


Stress exponent n in Northon law:

$$\dot{\varepsilon}_S = k\sigma^n$$

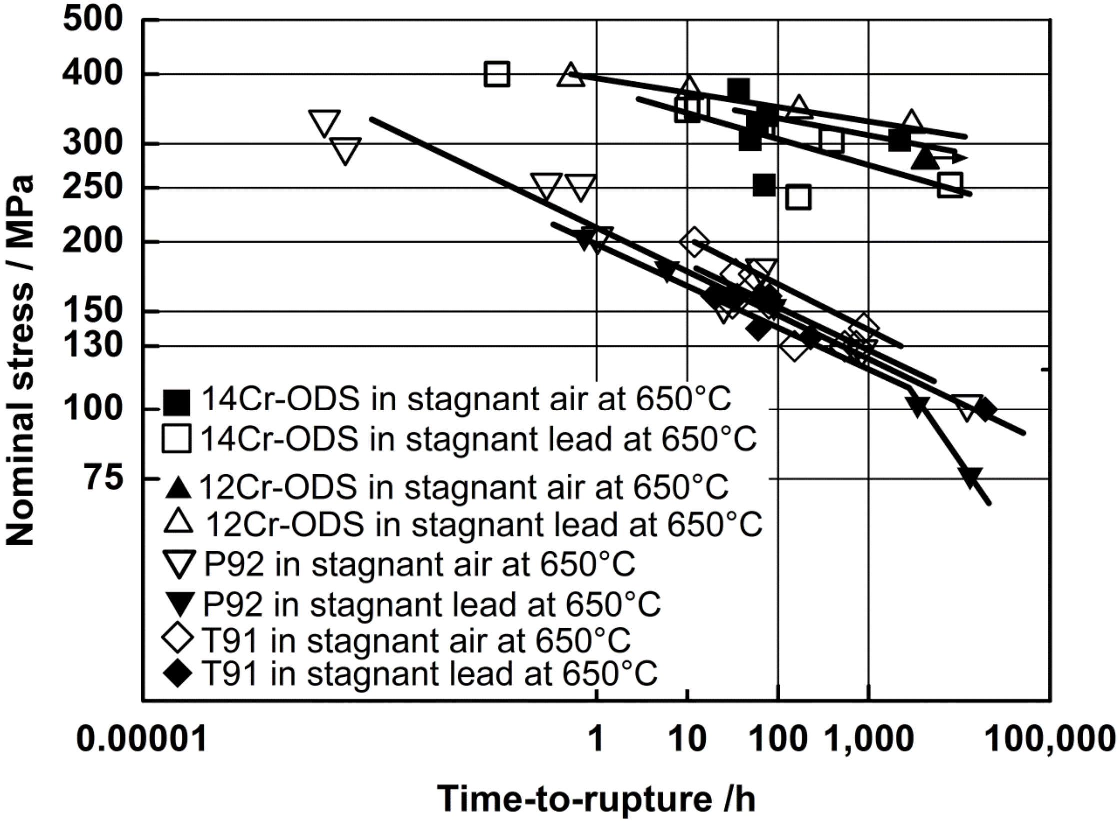
► No great difference in creep strength of T91 tested in both environments

Creep strength of 14Cr-1W and 12Cr-2W ODS steels in stagnant lead ($c_o=10^{-6}$ mass%) and air at 650°C



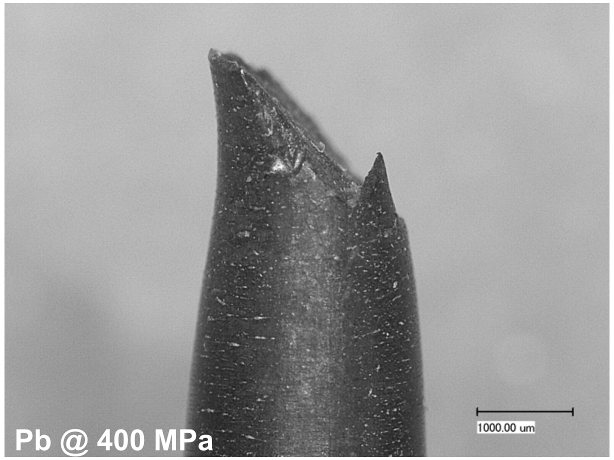
The 12-Cr ODS steel exhibits a slightly higher creep strength in stagnant Pb than the 14Cr-ODS steel

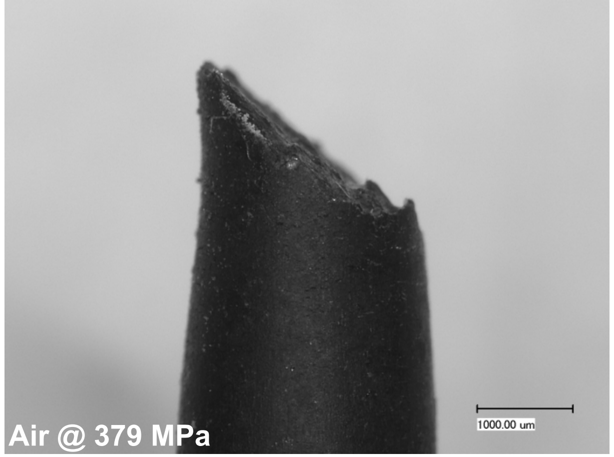
ODS steels against f/m steels T91 and P92, tested in air and lead at 650°C

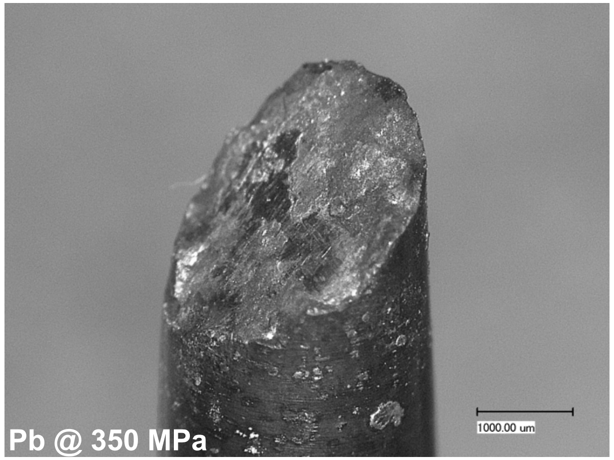


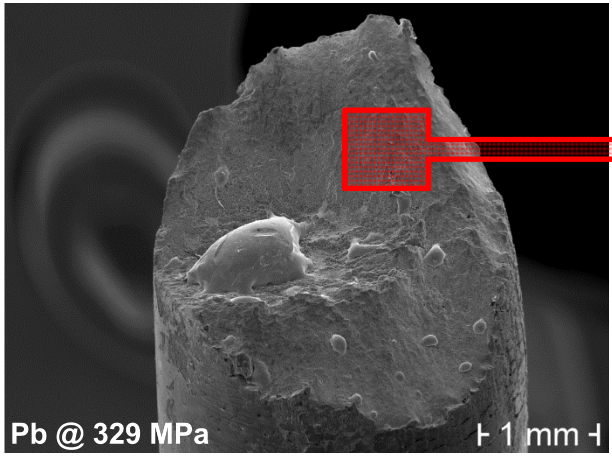
Creep-rupture strength of 12Cr- and 14Cr-ODS steels is factor 2.5 higher then the f/m steels until t_R=10,000 h and show no LME in contrast to P92 tested in Pb.

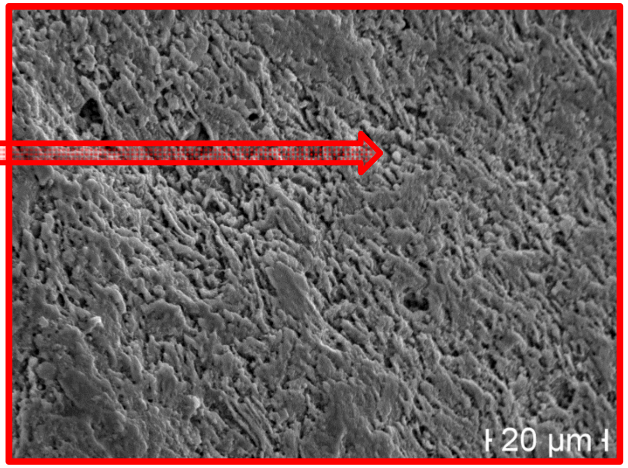
12Cr-ODS steels after creep-to-rupture tests





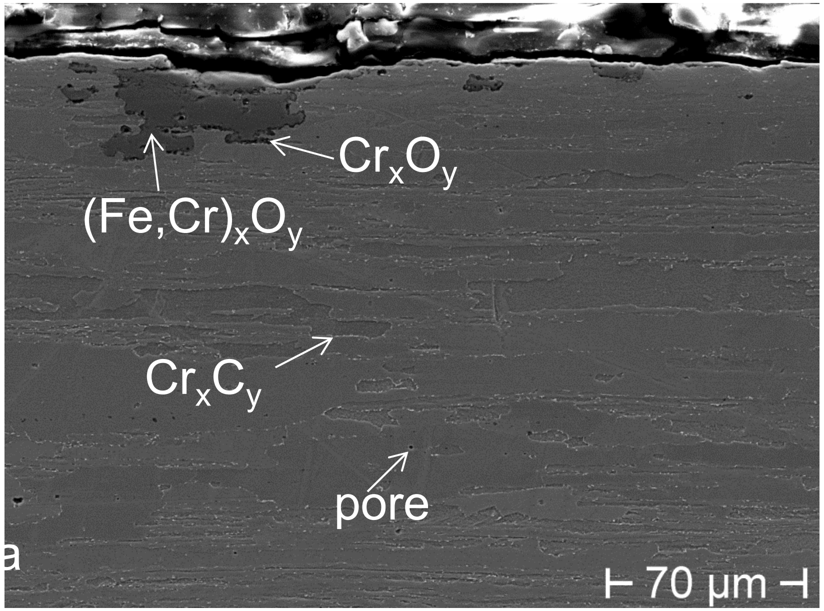


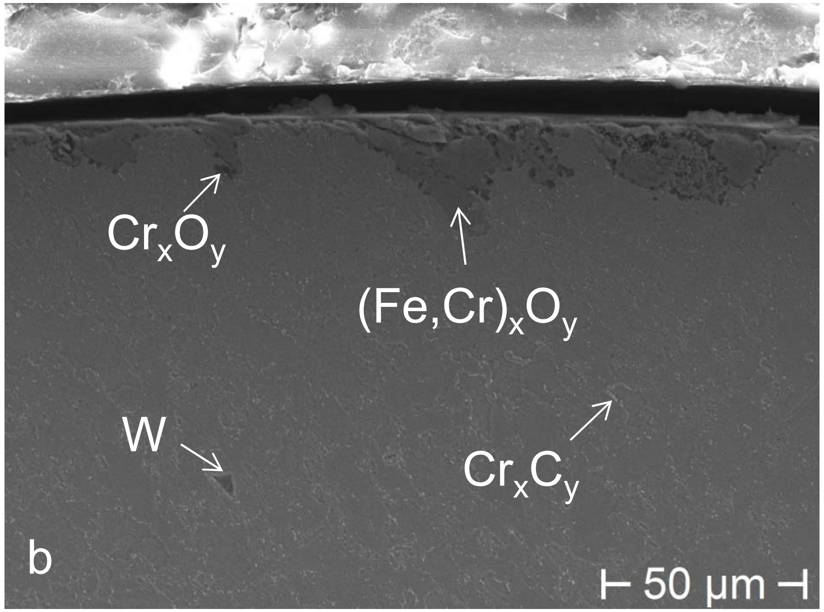




Shear fracture which is characteristic for ductile mode is proved by εc;R and Z
The higher stress, the higher $\varepsilon_{c;R}$ and Z were determined

12Cr-ODS steels after creep-to-rupture tests





Longitudinal (a) and perpendicular (b) cross-sections of the steel ruptured after t_R=2,982 h in Pb at 329 MPa

- **Oxide scale is irregular and contains Fe, Cr and O. The thickness is up to 30 µm.**
- **Until 2,982h exposure to Pb, no dissolution of the steel was observed**

Summary

- Heavy liquid metals (HLMs) are very appropriate coolants/targets for Nuclear (ADS, LFR) applications. Worldwide R&D has been established to buildup databases for compatibility issues of potential structural materials.
- F/M steels with 9%Cr show three stages of interaction with flowing LBE at 400-550°C, 10^{-7}-10^{-6} mass% dissolved oxygen, 2 m/s
  - Protective scaling – short term or local phenomenon (mainly at 400-450°C)
  - Oxidation – the general degradation mechanism
  - Direct liquid-metal attack – locally, after accumulation of liquid metal underneath the oxide scale
- Average rate of oxidation is lower
  - For higher Cr-content at 400-450°C
  - For fine-grained materials at 550°C
- Observed kinetics of oxidation is slower than parabolic
  - Corrosion rate increases by factor 2–3 for increase in temperature from 450 and 550°C
- Liquid metal attack shows
  - High local material loss in comparison to oxidation, e.g., increase by factor by factor 3–5 for T91 and 9%Cr-ODS at 550°C