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Karlsruhe Institute of Technology

Effect of oxygen concentration in static Pb at 700 °C and Pb-Bi eutectic at 550°C on corrosion behavior of aluminium-alloyed austenitic steels

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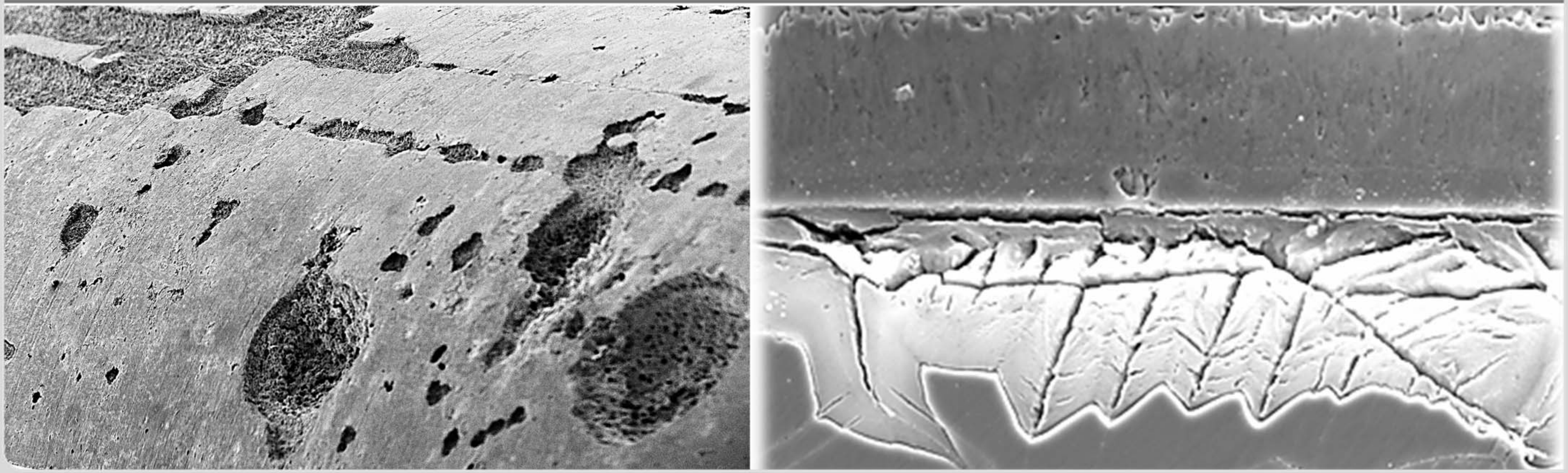
INSTITUTE FOR APPLIED MATERIALS – APPLIED MATERIALS PHYSICS (IAM-WPT)



Outline

- BACKGROUND ON COMPATIBILITY ISSUES OF STEELS IN CONTACT WITH HEAVY-LIQUID METALS AS APPLIED FOR GEN-IV REACTORS AND ADS

- EFFECT OF OXYGEN CONCENTRATION IN HEAVY-LIQUID METALS ON THE CORROSION RESPONSE OF ALUMINIUM-ALLOYED AUSTENITIC STEELS
  - *Corrosion test in static Pb at 700˚C*
  - *Corrosion test in static Pb-Bi eutectic at 550˚C*

- PRECIPITATIONS AND DEPOSITS FOUND IN THE CORRIDA LOOP AFTER OPERATION FOR 113000 h

- PHENOMENOLOGICAL MODEL DESCRIBING LIQUID-METAL EMBRITTLEMENT OF STEELS (LME) IN HEAVY-LIQUID METALS DEVELOPED IN PhMI NASU

LIQUID-METALS AS A FUNCTIONAL MEDIA FOR NOVEL REACTORS

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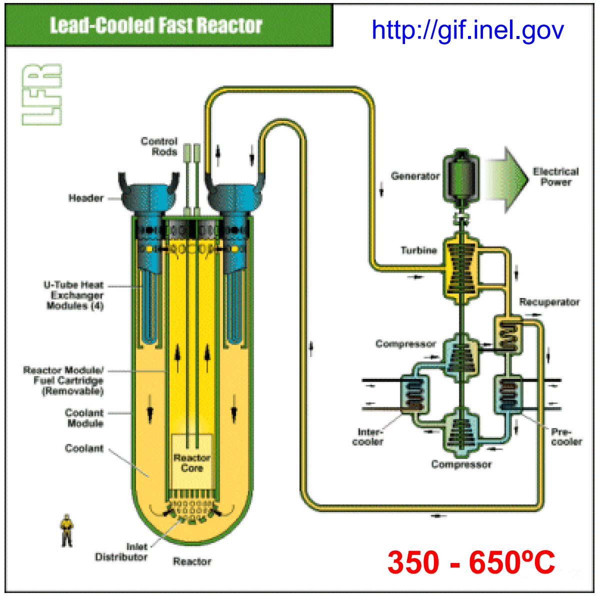
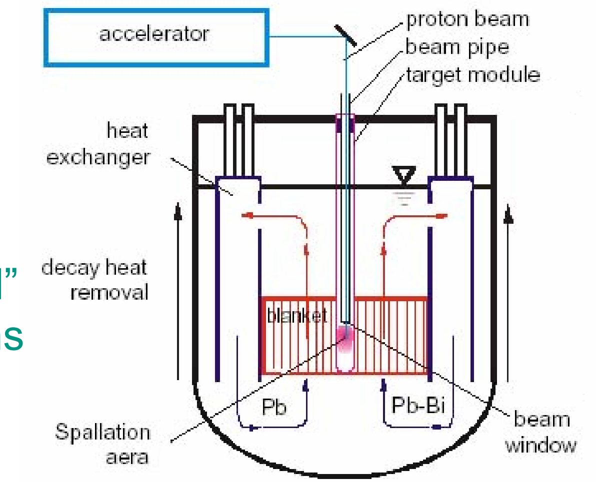
GENERATION IV



Accelerator Driven System



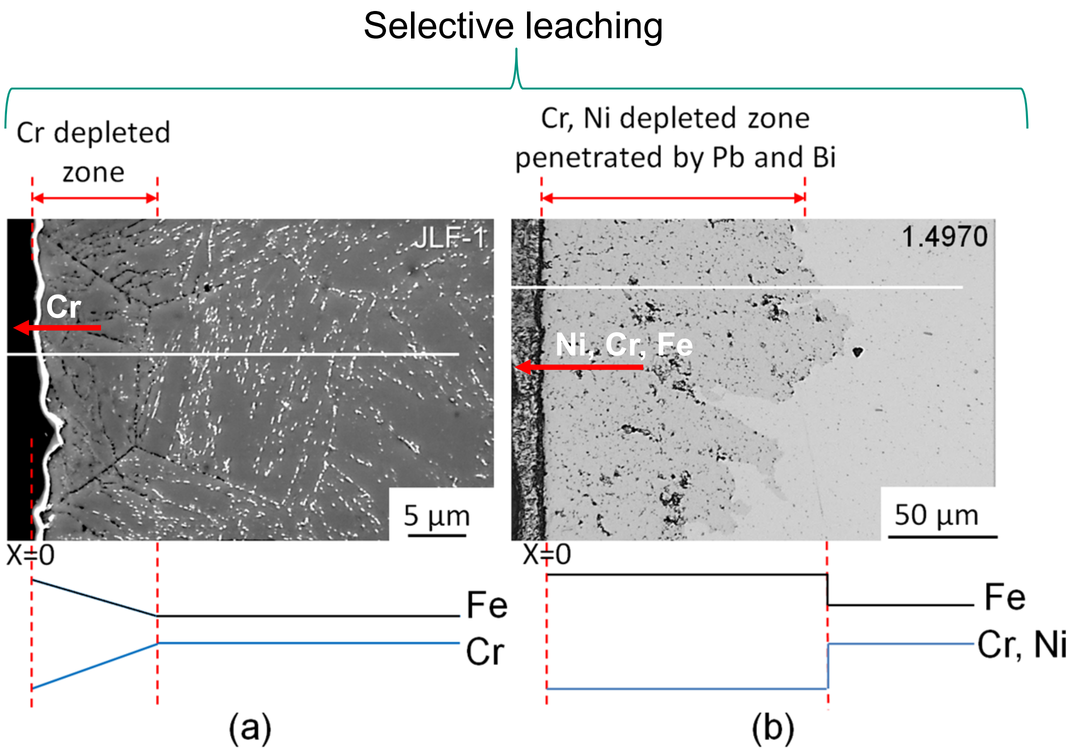
http://nucleartimes.jrc.nl

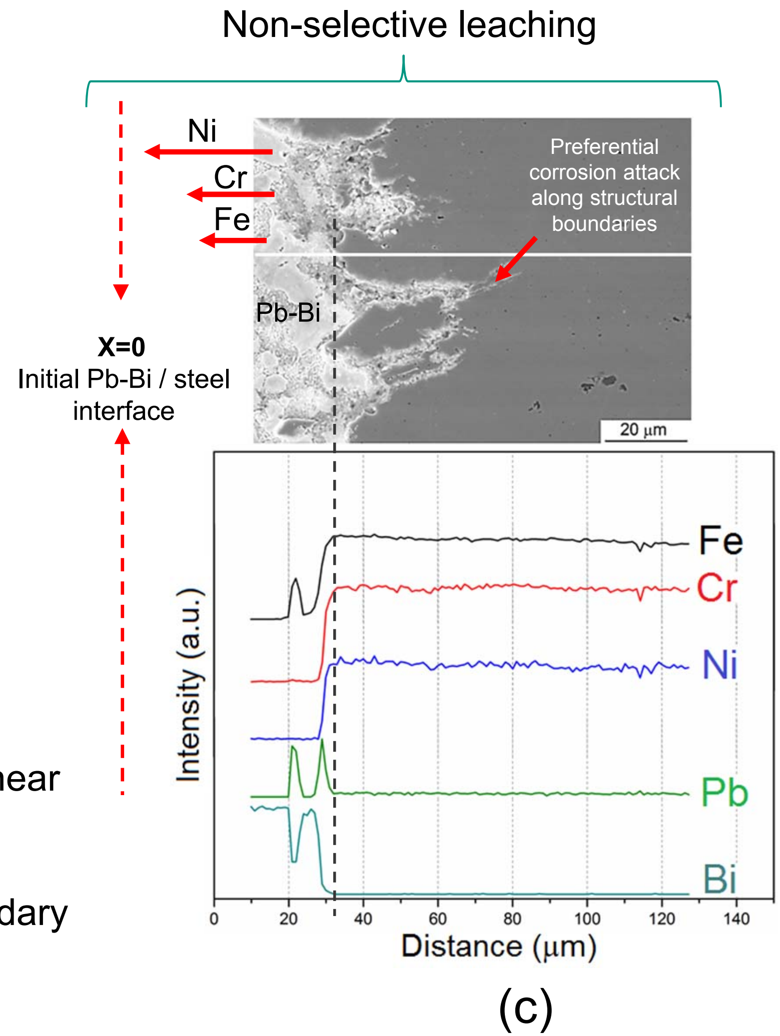
Accelerator Driven System

System “liquid metal / solid metal” – a common area of investigations

Concentrating Solar Power

DISSOLUTION OF STEELS IN HLM





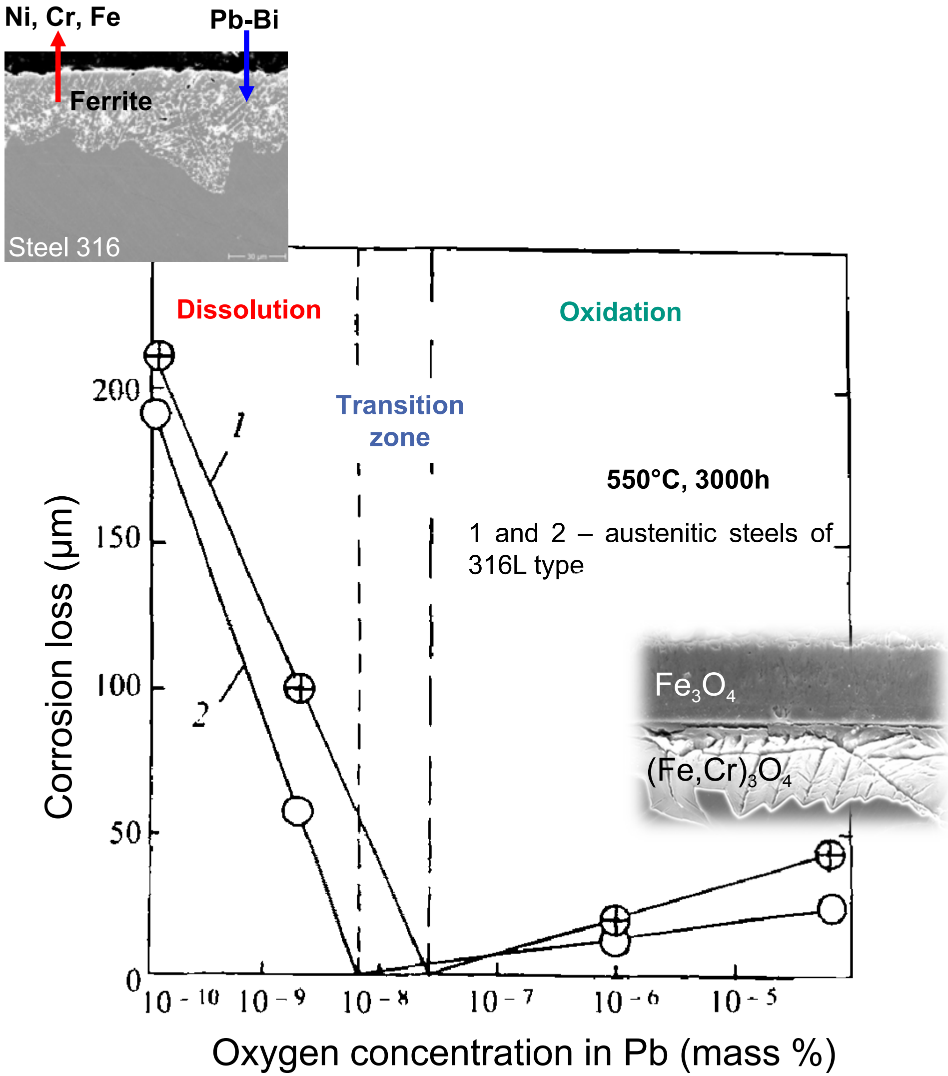
(a) Solution-based attack is controlled by the Cr diffusion in the near surface layer of steel;

(b, c) Solution-based attack is controlled by the diffusion in boundary layer of liquid metal.

BACKGROUND ON COMPATIBILITY ISSUES OF STEELS AND HEAVY-LIQUID METALS AS APPLIED FOR NOVEL REACTORS

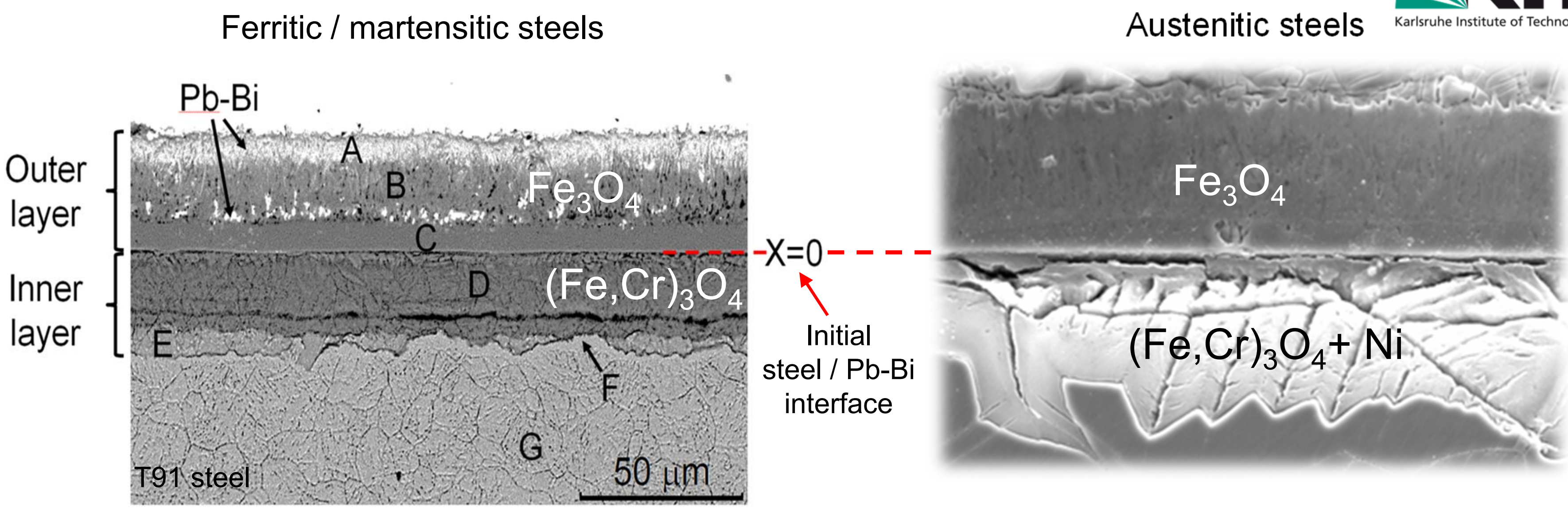
Main corrosion modes

1. **Dissolution**
   - Leaching of Ni, Cr and Fe
2. **Oxidation**
   - Formation of Fe-based scale



I.V. Gorynin et al. Met. Sci. Heat Treat. 41 (9) (1999) 384–388

OXIDATION OF STEELS IN HLM



Composition of bi-layer scale

- X=0 – initial steel / HLM interface
- Outer layer – Fe_3O_4
  - A + B – sub-layers with loose columnar structure containing Pb and Bi
  - C – sub-layer with compact equi-axial structure
- Inner layer
  - D – $Fe(Fe,Cr)_2O_4$ spinel
  - E – inner oxidation zone (Fe + Cr_2O_3)
  - F – Cr-depleted zone
- Steel T91 – G

- Bi-layer scale, with outer Fe_3O_4 (magnetite spinel) and inner $Fe(Fe,Cr)_2O_4$ spinel-type oxide layers, typically forms on the surface of steels in contact with oxygen-containing Pb and Pb-Bi melts
- Growth of scale is governed by the outward diffusion of iron cations
- Inward growth of Fe-Cr spinel at the oxide/steel interface could be accessed from the ***dissociative growth theory***: vacancies generated by outward diffusion of iron cations precipitate at the oxide/steel interface forming cavities (pores) into which the oxide dissociates with evaporating oxygen providing further oxidation of steel (S. Mrowec, Corrosion Science 7 (1967) 563-578).

EXAMPLE OF SCALE EVOLUTION WITH TIME

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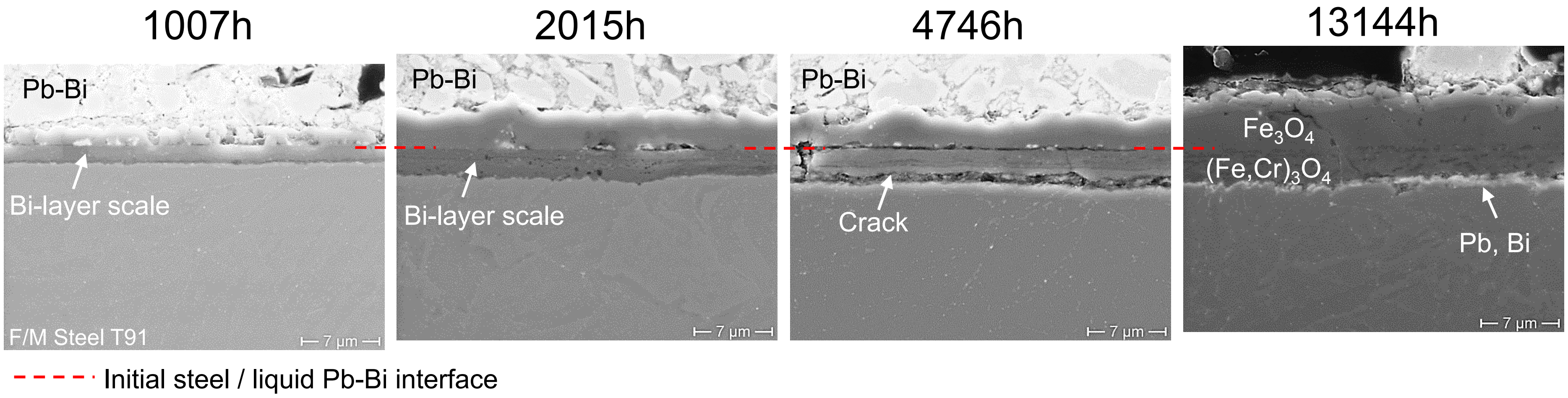
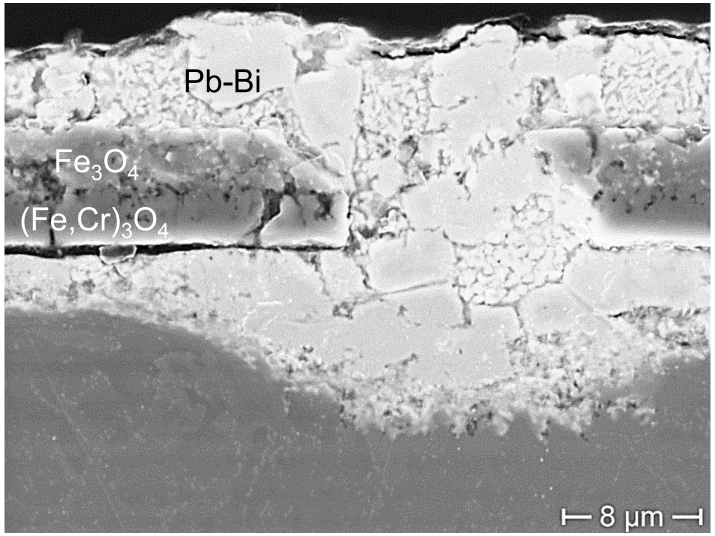
Flowing Pb-Bi (2 m/s), 10^{-7} mass%O, 400°C



Dissolution attack as a result of scale failure

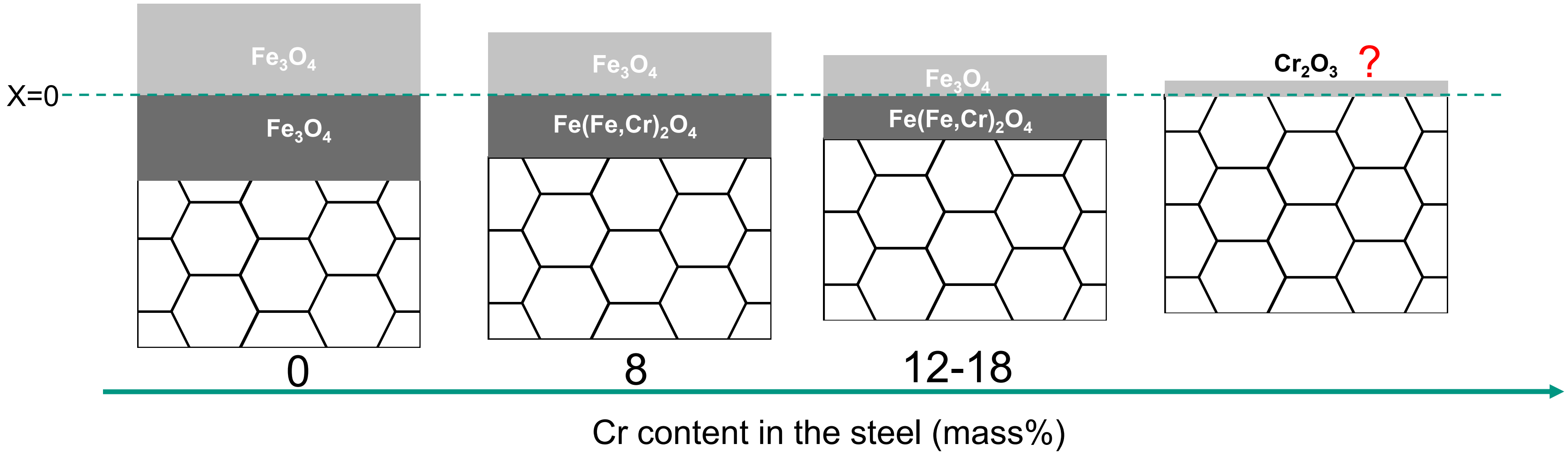


- Degradation of scale with time results in initiation of local dissolution attack
- Re-healing of scale does not take place
- The long-term viability of protective scale on the surface of steels facing Pb melts is one of the main task for successful application of oxygen-controlled Pb melts

EFFECT OF ALLOYING ON OXIDATION IN Pb MELTS

Cr-alloyed steels

Effect of Cr content on scale composition, morphology and thickness



- Conditions: T≤570°C, $C[O]_{HLM} > Fe_3O_4$
- Thickness of bi-layer scale decreases with increasing Cr content in the material
- Formation of single-layer Cr-rich oxide film is a short-time period (incubation)

EFFECT OF ALLOYING ON CORROSION

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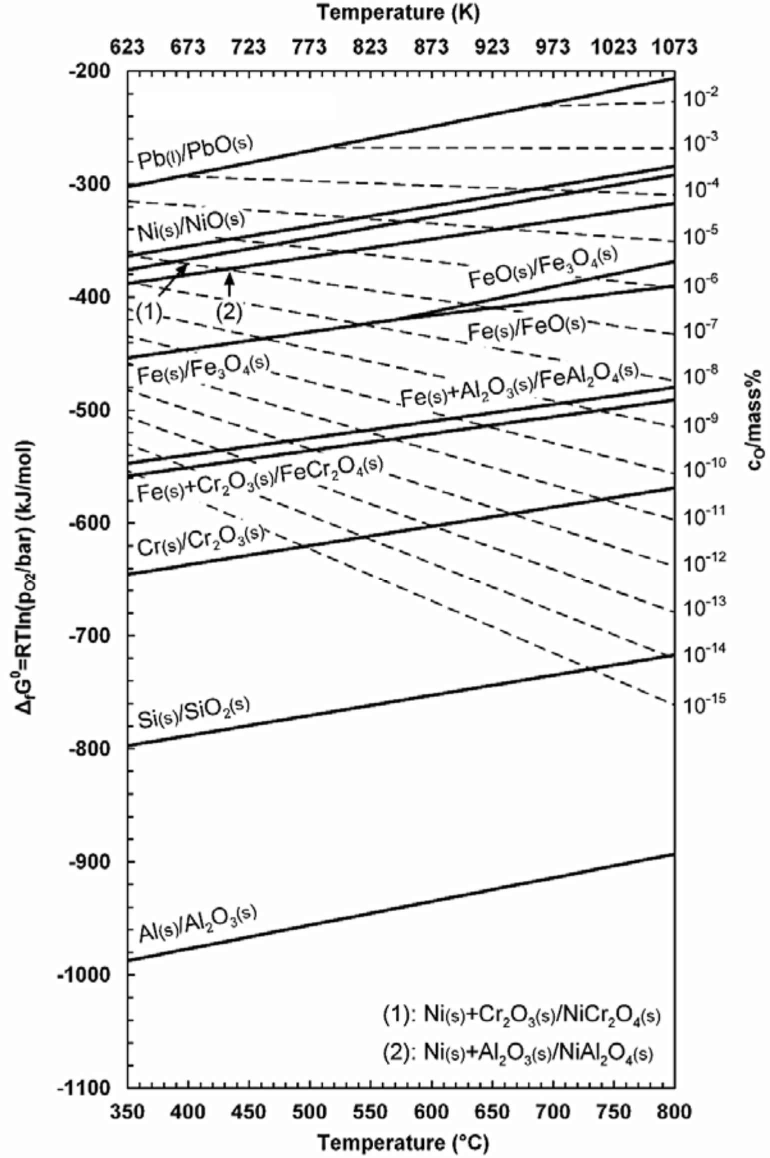
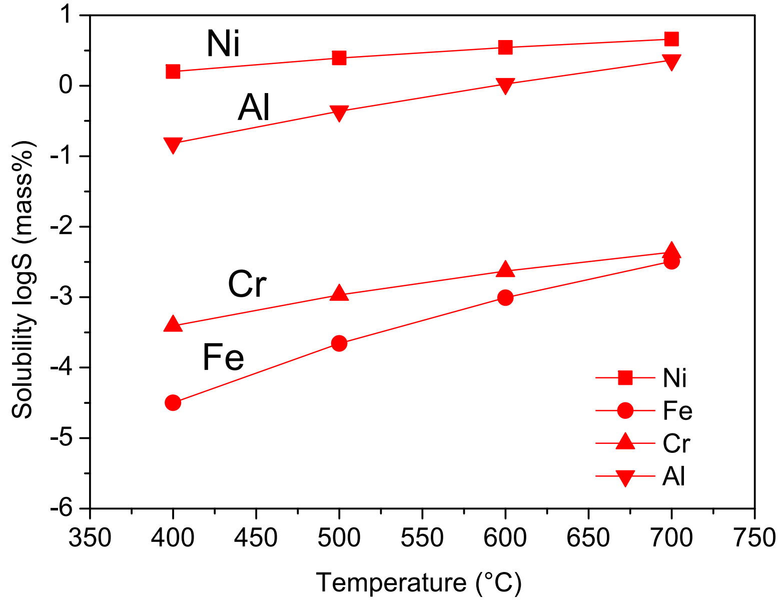
Effect of alloying on oxidation



Effect of alloying on dissolution



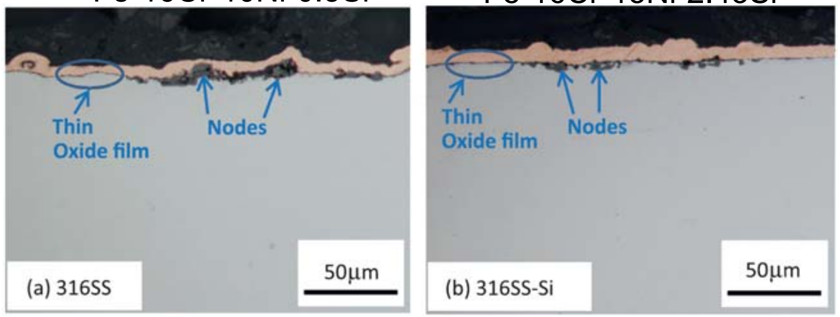
Solubility of pure metals in LBE as a function of temperature

- Transfer from Fe-based bi-layer scale to single layer oxide films based on Cr, Si, Al is highly desirable
- Alloying elements in steels which might improve oxidation resistance are typically highly soluble in HLM

Silicon alloyed steels

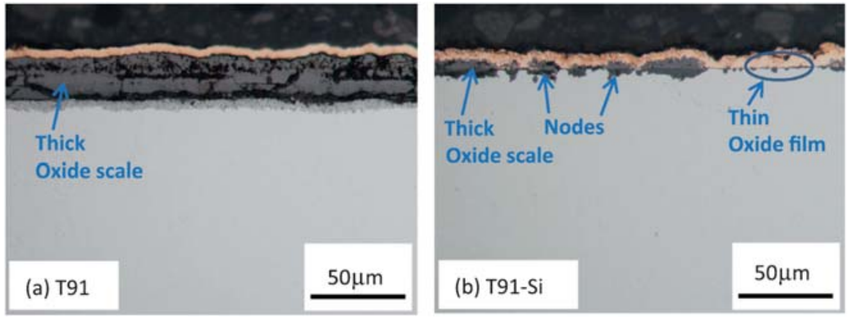
Silicon-alloyed austenitic steels

Fe-16Cr-10Ni-0.5Si   Fe-16Cr-15Ni-2.43Si



Silicon-alloyed F/M steels

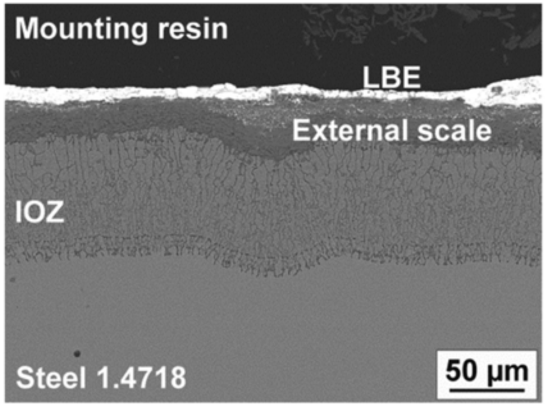
Fe-8Cr-0.4Si   Fe-8Cr-1.45Si



Yuji Kurata / Journal of Nuclear Materials 437 (2013) 401–408

- Static Pb-Bi at 550°C with 10^{-5} mass%O, ~1300h
- The Si addition reduces the scale thickness under the high oxygen condition
- The Si addition has no significant effect under the low oxygen condition (10^{-8} mass%O)

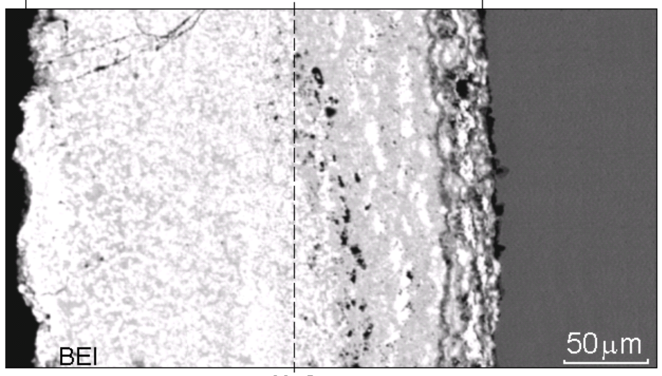
Fe-9Cr-3Si



- Flowing LBE, 550°C, 10^{-6} mass%O, 15028 h
- Intensive development of IOZ after long-term exposure

C. Schroer et al. / Journal of Nuclear Materials 469 (2016) 162–176

Scale   Fe-13Cr-3Si



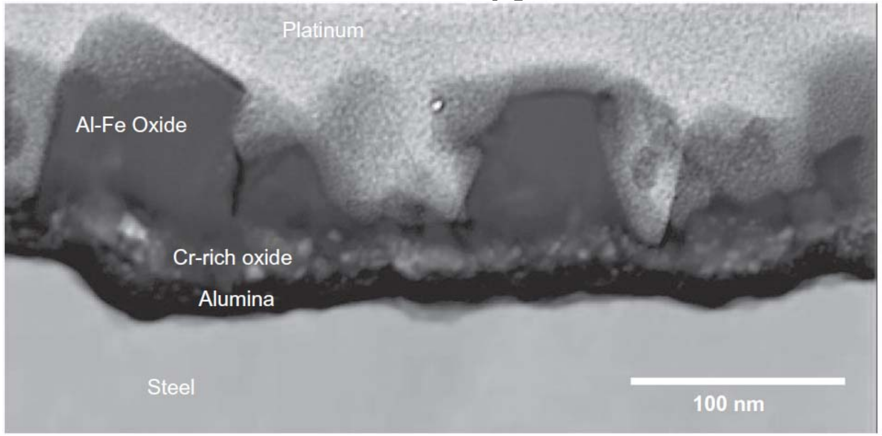
X=0

- Static Pb, 650°C, 10^{-3} mass%O, 50 h
- Extreme oxidation with formation of non-protective thick scale totally penetrated by Pb

V. Tsisar / PhD (2005)

Aluminum-alloyed F/M steels

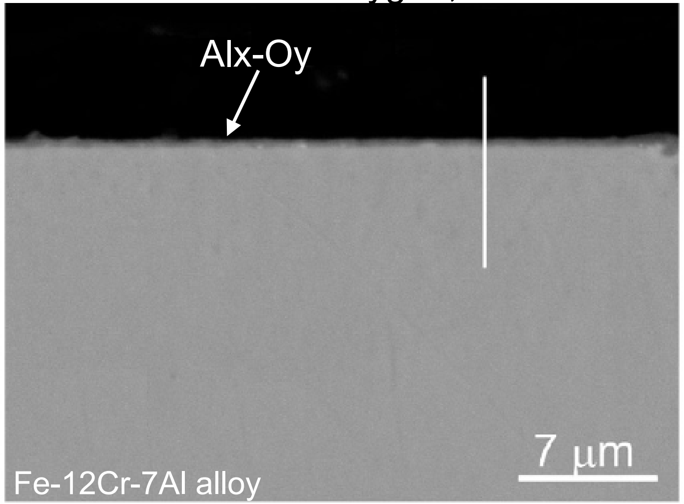
Static Pb, Fe-10Cr-6Al-RE alloy, 550 °C, 10^{-7} wt.% oxygen, 10000h

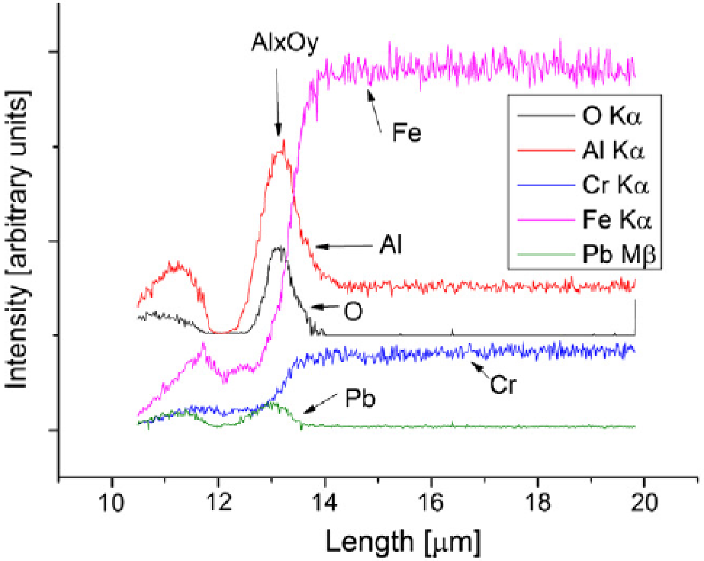


J. Ejenstam, P. Szakálos /Journal of Nuclear Materials 443 (2013) 161–170

- Thin Al-rich oxide layer, formed at the metal–oxide interface, prevented Pb penetration into the bulk steel at least up to 10000 h
- Synergetic effect of Cr and Al is important for formation of protective oxide film
- **Preliminary results shows a potential for using Al-alloyed steels at higher temperatures (>500 °C)**
- **Tests in dynamic HLM are necessary** to investigate viability of alumina films and it self-healing abilities

Static Pb, Fe-12Cr-7Al alloy, 600 °C, 10^{-6} wt.% oxygen, 1830h





A. Weisenburger et al. / Journal of Nuclear Materials 437 (2013) 282–292

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CHEMICAL COMPOSITION AND STRUCTURE OF AUSTENITIC STEELS ALLOYED BY ALUMINIUM

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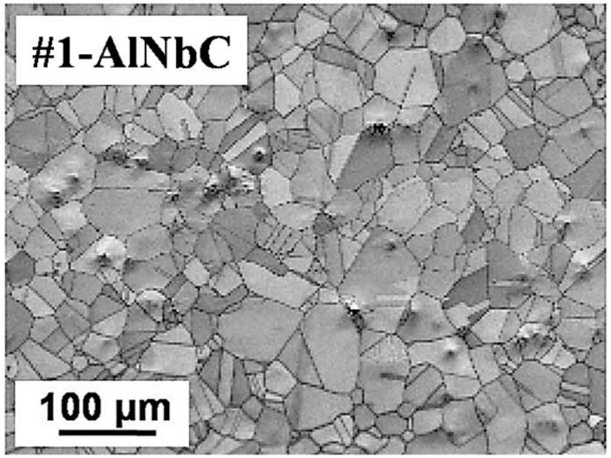
- Alumina-Forming Austenitic (AFA) stainless steels with improved creep resistance (strengthening with Laves phases and carbides) and oxidation resistance due to formation of Al_2O_3 at high temperatures in gaseous media are under developing (Y. Yamamoto et al., Metall and Mat Trans A 42 (2011) 922–931)
- **Applicability of AFA steels in Pb and Pb-Bi arouses interest and requires experimental investigations !**

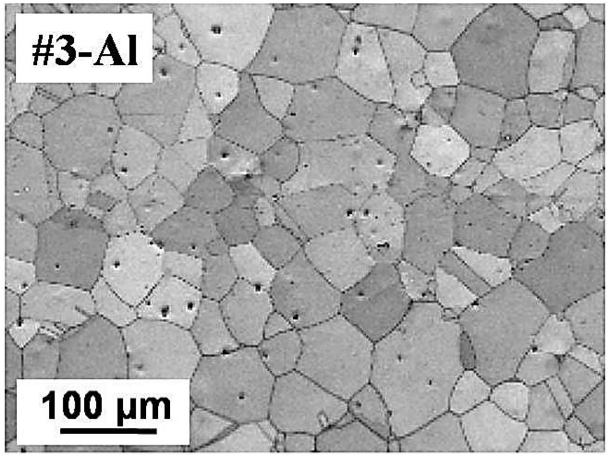
(Fe–Bal.)	Cr	Ni	Mo	Mn	Si	Al	Nb	C
# 1-AlNbC	11.7 (±0.02)	18.0 (±0.02)	1.99 (±0.003)	0.0887 (±0.0003)	0.401 (±0.0006)	2.32 (±0.008)	0.577 (±0.003)	0.0086 (±0.0003)
# 3-Al	11.7 (±0.02)	18.0 (±0.05)	2.00 (±0.007)	0.118 (±0.0005)	0.377 (±0.0009)	2.90 (±0.010)	<0.001	0.0300 (±0.0006)

Fe-18Ni-12Cr-AlNbC



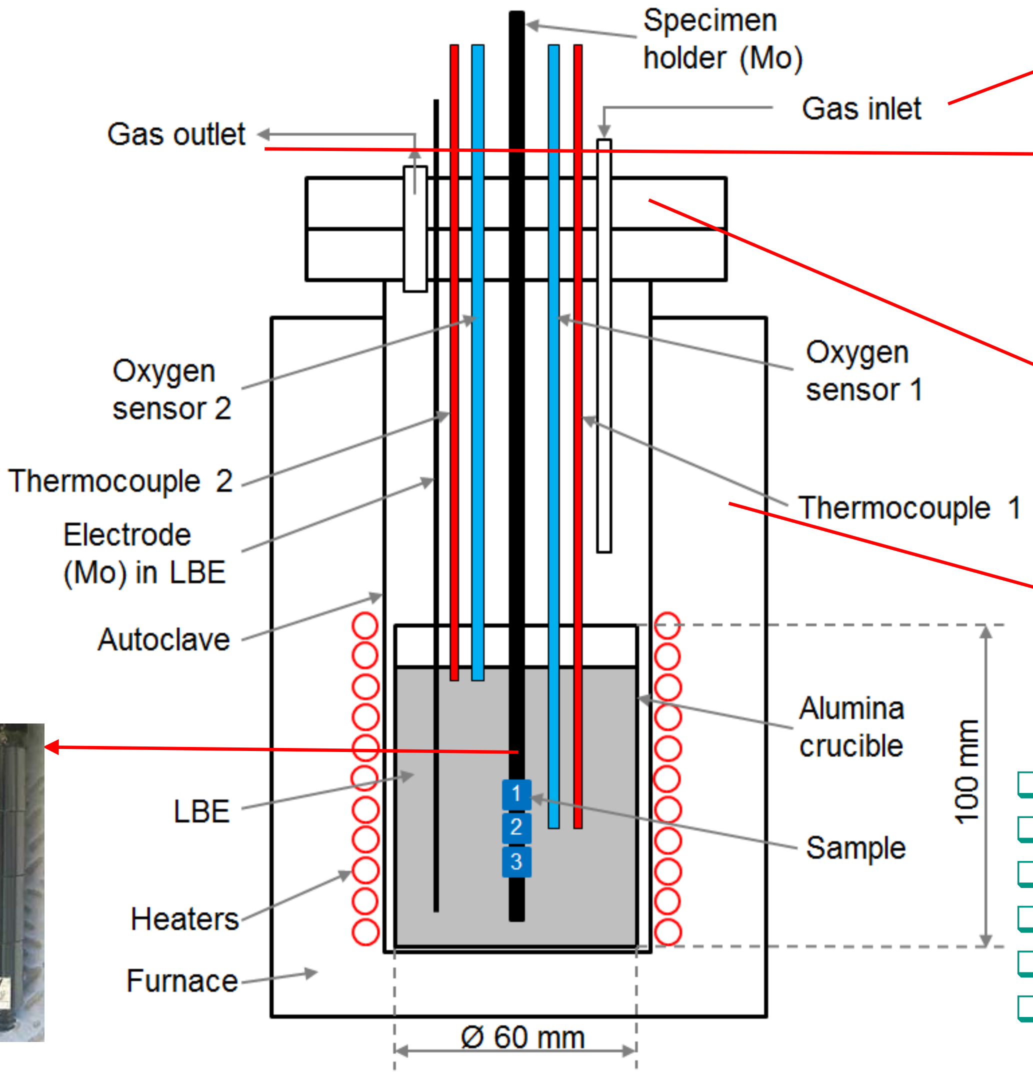
Grain size

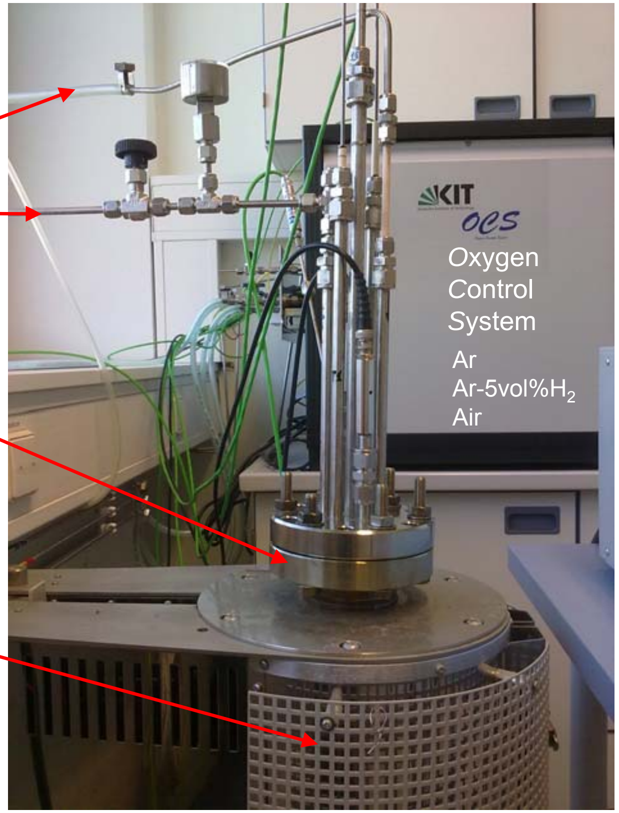
Fe-18Ni-12Cr-Al



School of Material Science and Engineering, University of Science and Technology Beijing, Beijing 100083, PR China

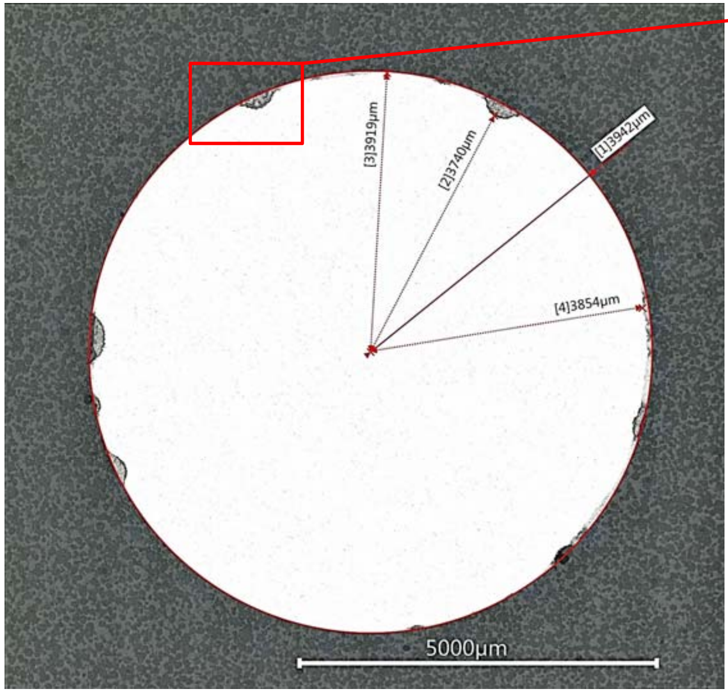
APPARATUS FOR STATIC CORROSION TESTS IN HEAVY LIQUID METALS

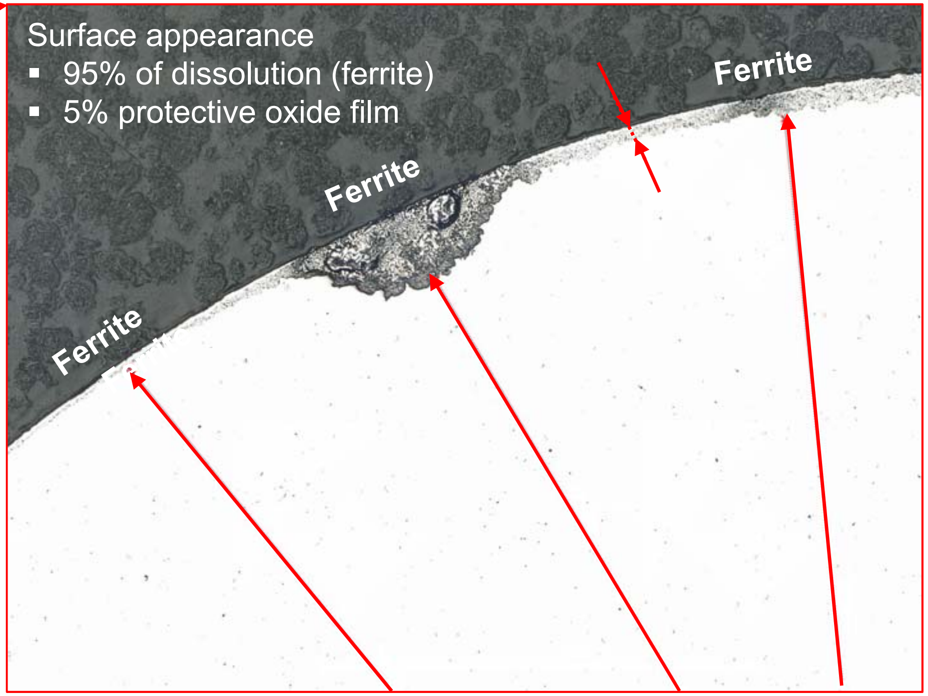




- ~2kg HLM (Pb, Pb-Bi, Sn)
- Working temperatures up to 700 ºC
- Ar+5%H2 / Ar / Air gas mixture above melt
- Two Pt/Air oxygen sensors
- Oxygen control system
- Cylindrical samples (Ø8x10 mm)

POST-TEST QUANTIFICATION OF CORROSION LOSS USING METALLOGRAPHIC METHOD FOR CYLINDRICAL SPECIMENS





1. Measurement of initial diameter in a laser micrometer (0.1 µm resolution)
2. Measurement of post-test diameter (12th measurements with rotation angle 15°) or radius of unaffected material on the cross-section
3. Measurement of thickness of corrosion zones (1 µm resolution)
4. % of occurrence of different corrosion modes
5. Extra measurements for determination of maximum depth of corrosion attack

Example of 3D light-optical topography of surface for measurement of depth of local corrosion attack directly from the surface

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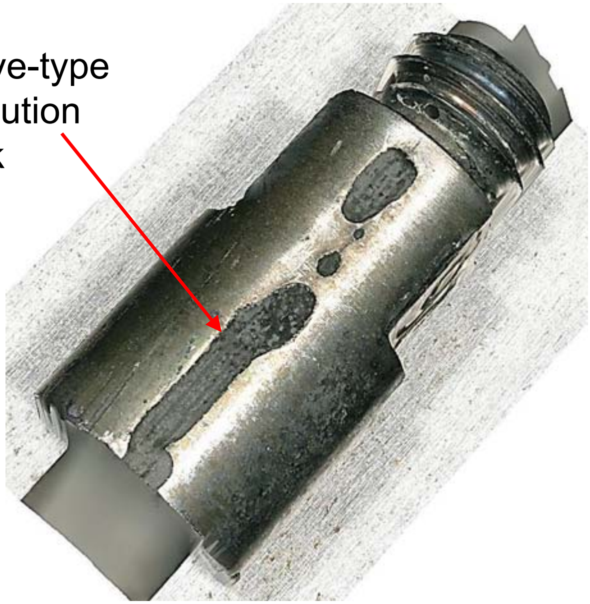
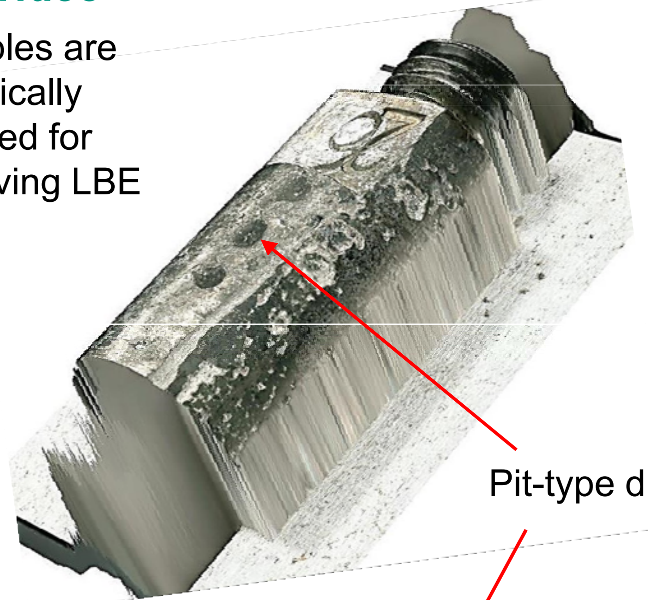
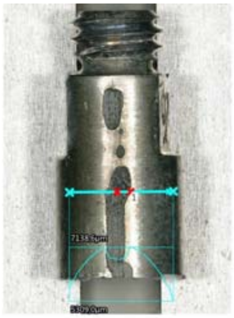
Groove-type dissolution attack



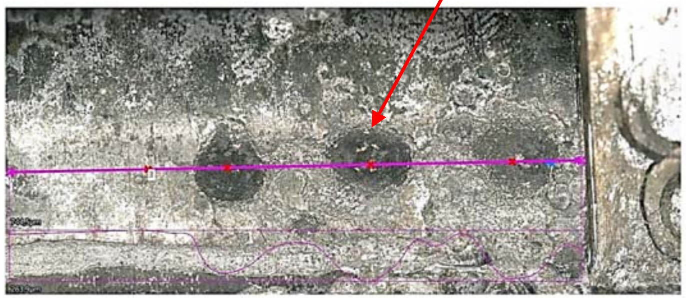
- Samples are chemically cleaned for removing LBE

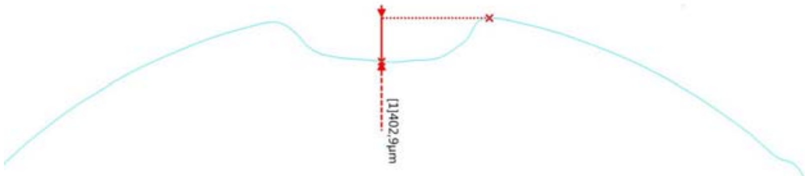


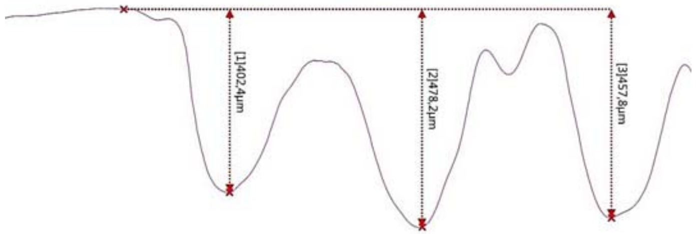
Pit-type dissolution attack



- Depth of local attack
- Area of severe corrosion attack







Cleaning of samples from solidified rests of HLM is a prerequisite for surface topography!

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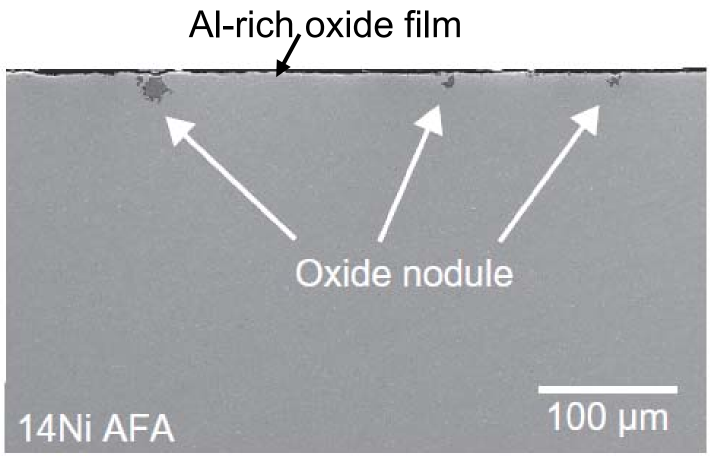
Scarce literature data

J. Ejenstam, P. Szakálos.
Journal of Nuclear Materials 461 (2015) 164–170

Fe-14Cr-14Ni-2.5Al-1.6Mn-2.5Mo-0.9Nb



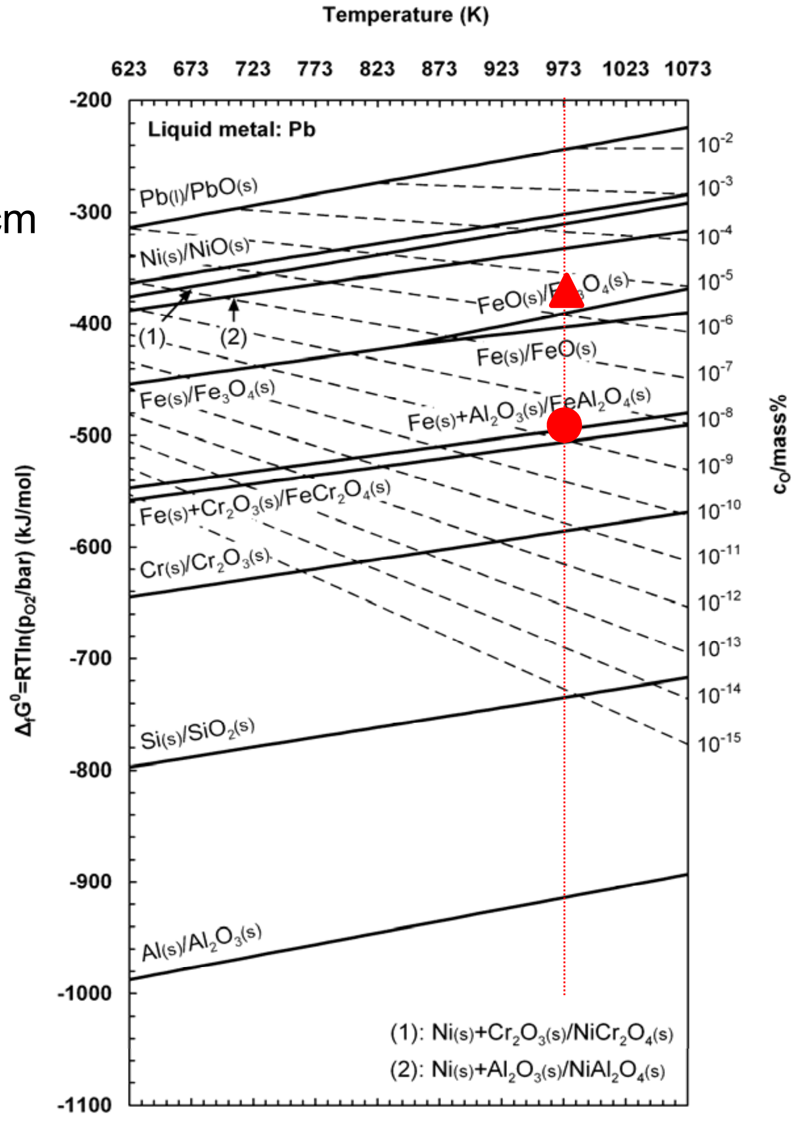
- Static Pb
- 550°C
- 10^{-7} wt.% oxygen
- one year

- Thin (<100 nm) protective Al-rich oxide film was formed on the14Ni AFA alloy after one year indicating that this alloy is a potential candidate for use in Pb-cooled reactors
- **Tests in flowing oxygen-controlled HLM are of interest** to show the long-term viability of protective oxide film on the alloy surface
- **High-temperature tests are of interest** as well

CORROSION TESTS IN STATIC Pb

Test conditions

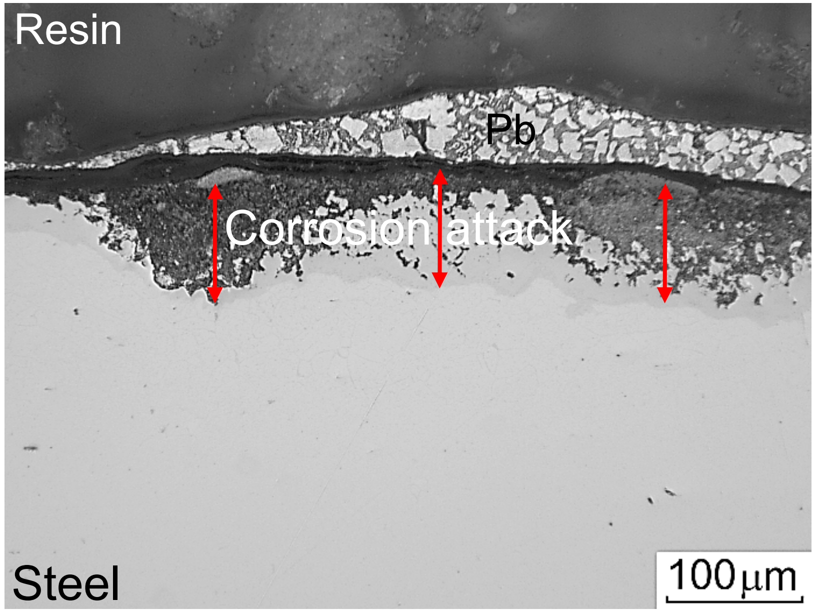
- Constant:
  - volume of Pb = 2 kg
  - ratio of Pb volume to surface of samples is 25 cm
  - temperature 700°C
  - exposure time ~1000 h
- Varying oxygen concentration in Pb
  - ● Test 1: 5×10^{-9} mass%O
  - ▲ Test 2: $\geq10^{-6}$ mass%O



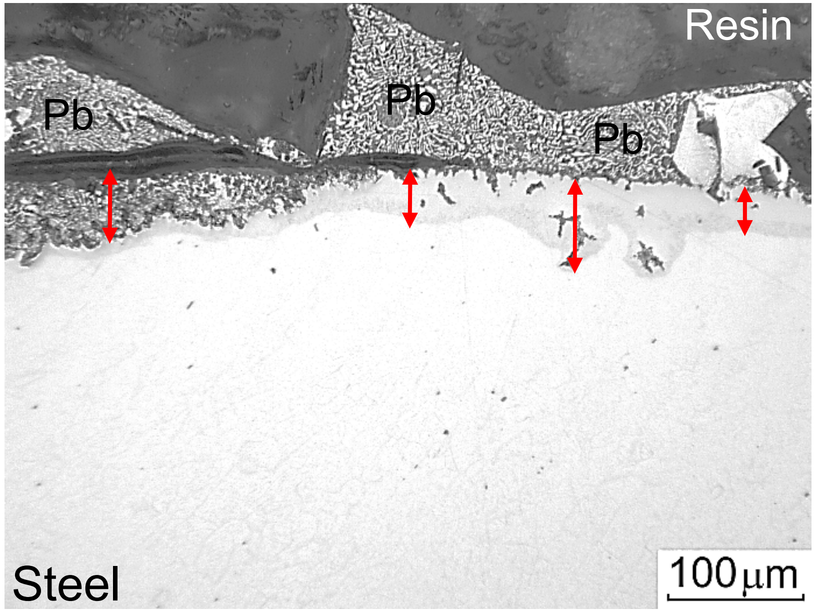
Dependence of the thermodynamic stability of selected oxides of steel constituents on temperature and the concentration of oxygen dissolved in liquid Pb
[C. Schroer, J. Konys. FZKA 7364]

Appearance of corrosion attack after test 1 - 5×10^{-9} mass%O

Steel Fe-18Ni-12Cr-AlNbC

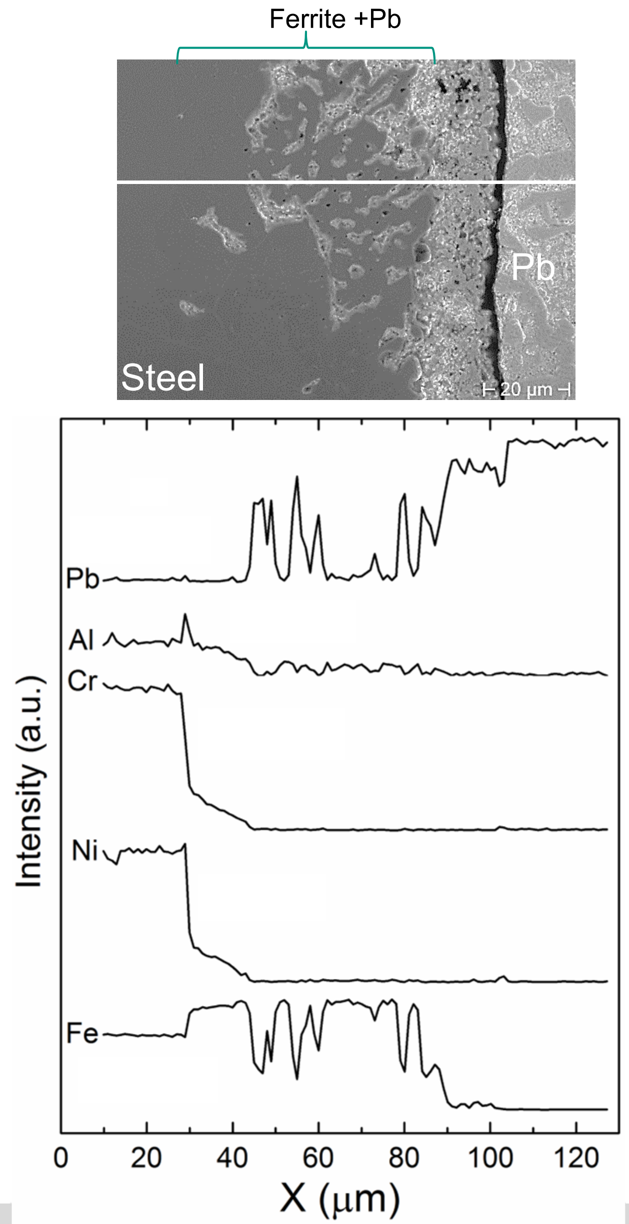


Steel Fe-18Ni-12Cr-Al

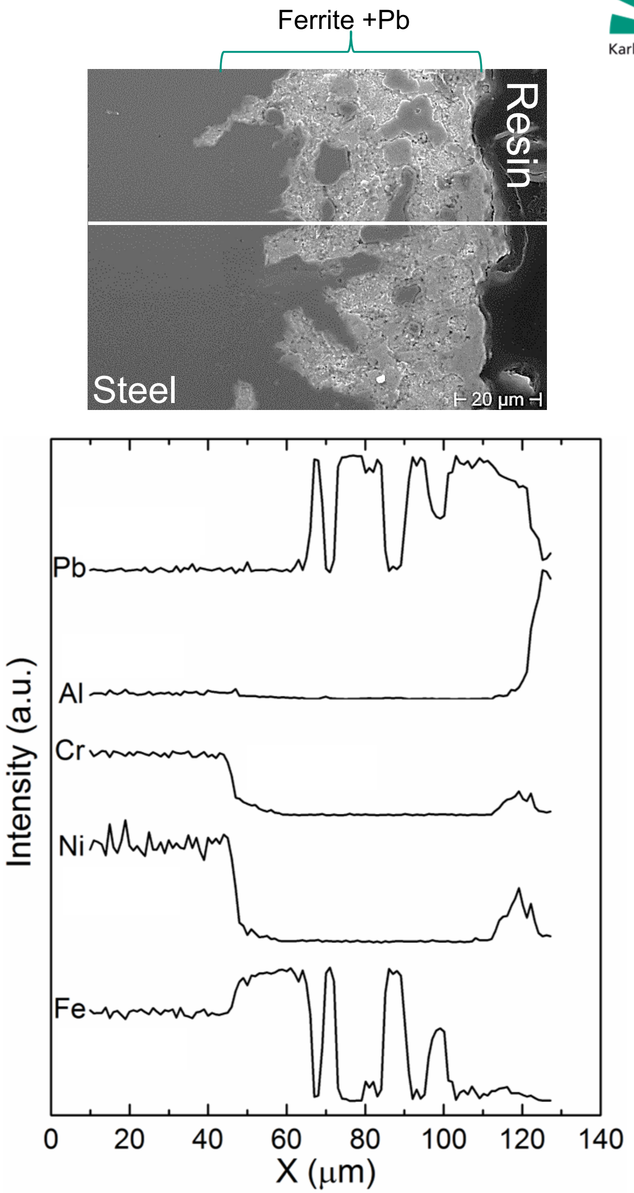


- Dissolution is a dominating (general) corrosion mode

Steel Fe-18Ni-12Cr-AlNbC



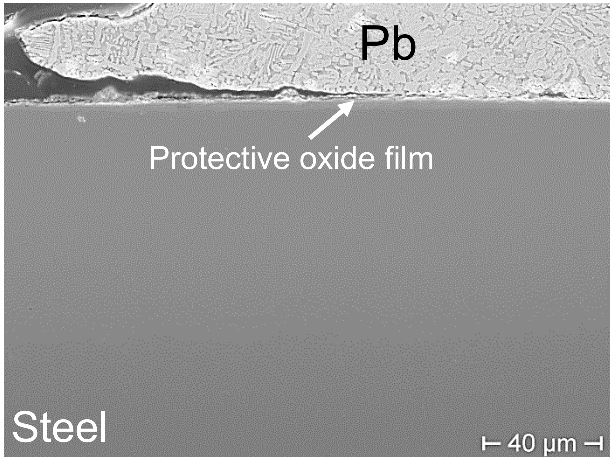
Steel Fe-18Ni-12Cr-Al



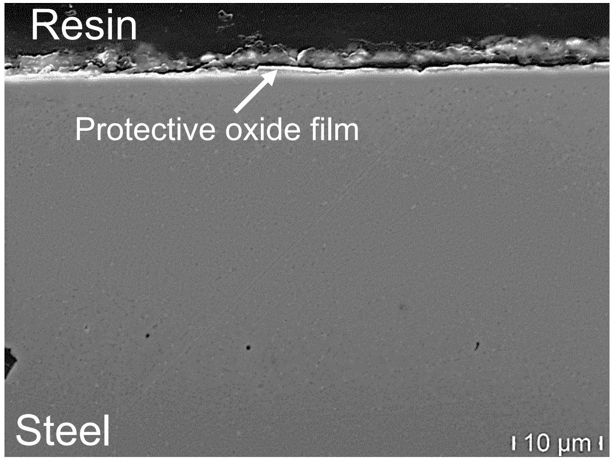
Appearance of corrosion attack after test 2 - ≥10^{-6} mass%O

General corrosion trend – protective oxide film

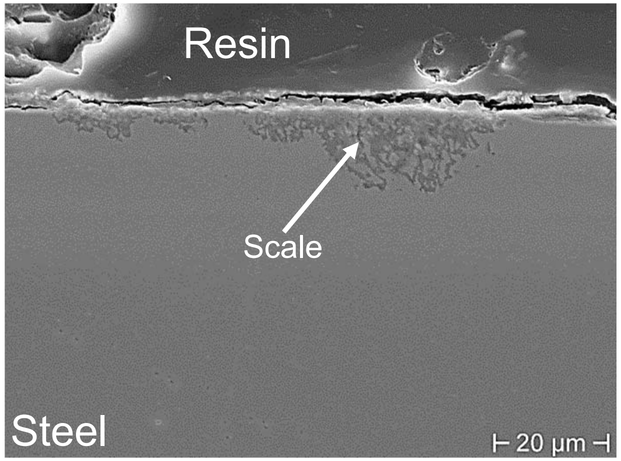
Steel Fe-18Ni-12Cr-AlNbC

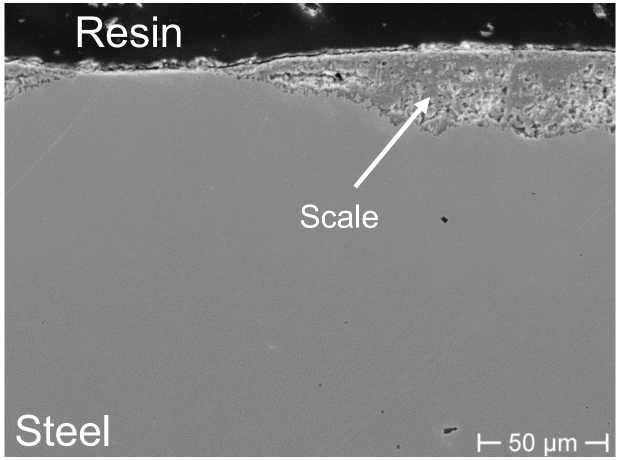


Steel Fe-18Ni-12Cr-Al



Local corrosion trend - scale formation

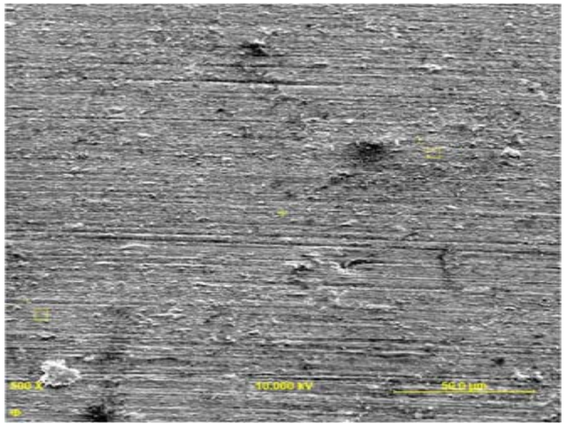


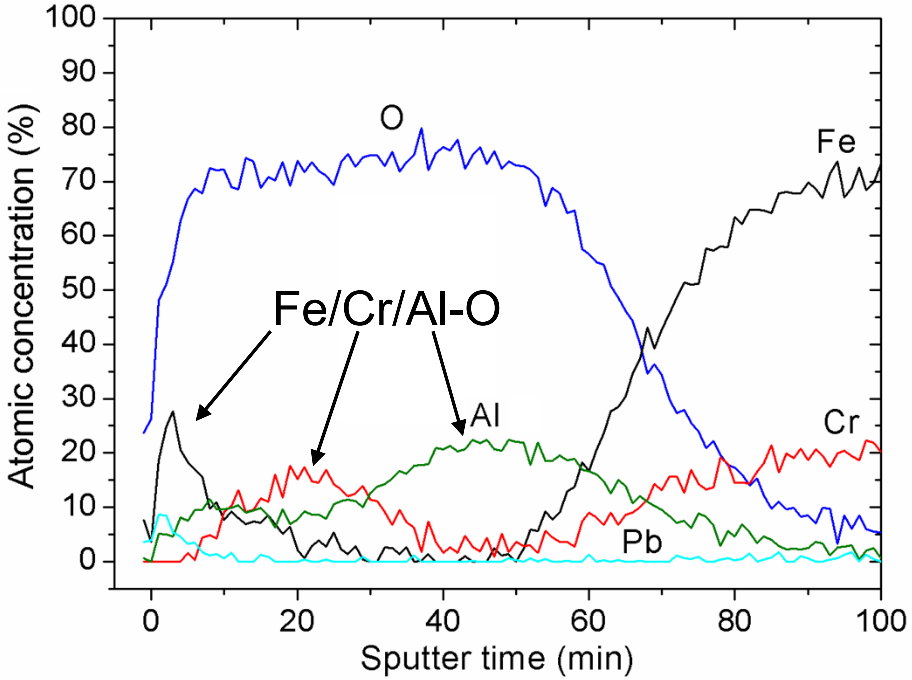


Steel Fe-18Ni-12Cr-AlNbC

General corrosion trend:
Protective scale formation

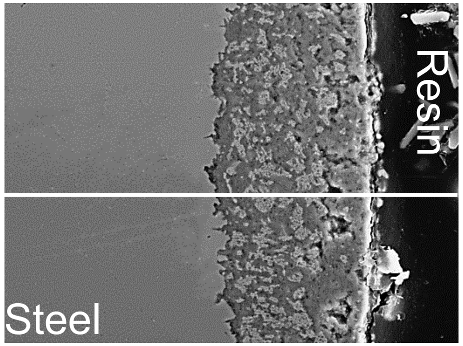
Sample surface

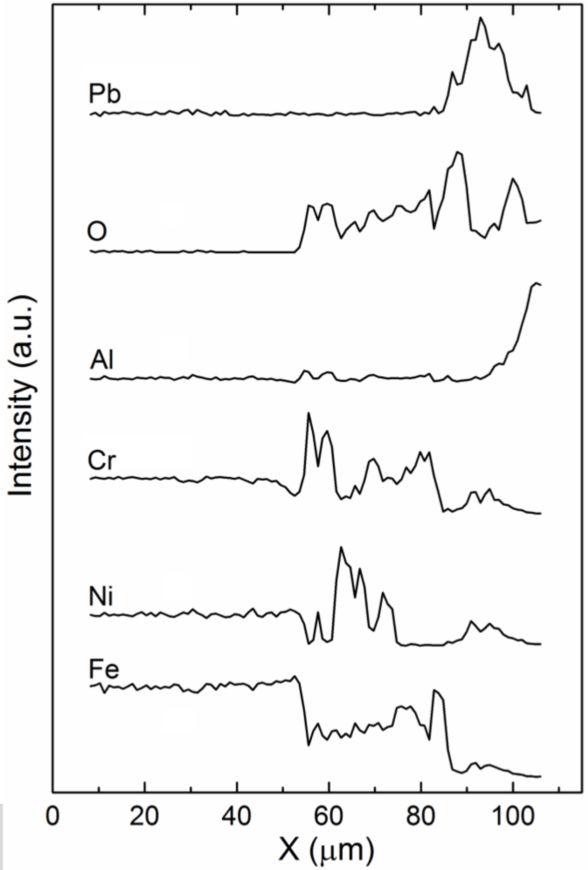




Steel Fe-18Ni-12Cr-Al

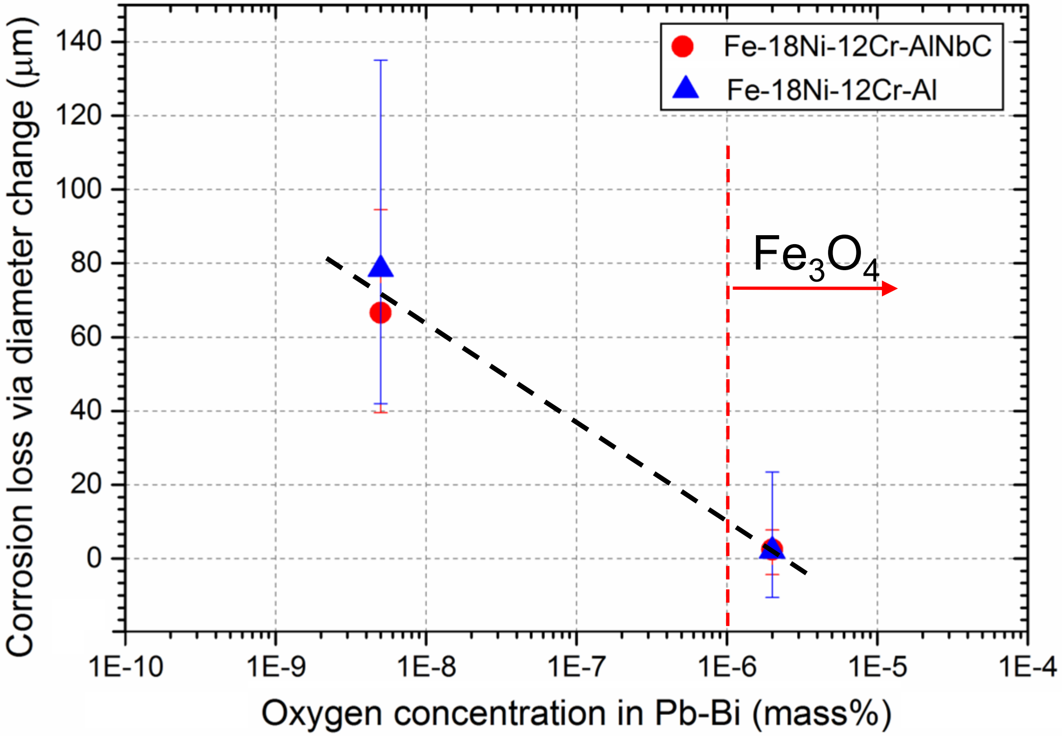
Local corrosion trend:
Scale formation





QUANTIFICATION OF CORROSION LOSS

Static Pb, 700˚C,1000h



- With increase in oxygen concentration in Pb the corrosion mode changes from dissolution to oxidation resulting in substantial decreasing in corrosion loss

- BACKGROUND ON COMPATIBILITY ISSUES OF STEELS AND HEAVY-LIQUID METALS AS APPLIED FOR NOVEL REACTORS

- **EFFECT OF OXYGEN CONCENTRATION IN HEAVY-LIQUID METALS ON THE CORROSION RESPONSE OF ALUMINIUM-ALLOYED AUSTENITIC STEELS**
  - *Corrosion test in static Pb at 700˚C*
  - *Corrosion test in static Pb-Bi eutectic at 550˚C*

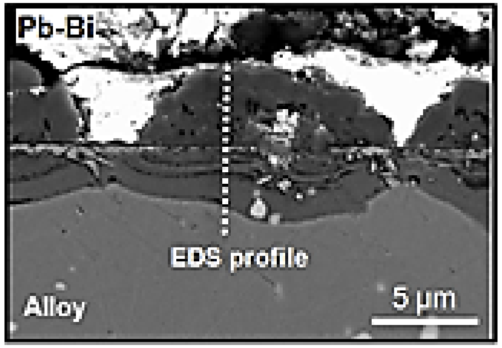
- PRECIPITATIONS AND DEPOSITS FOUND IN THE CORRIDA LOOP AFTER OPERATION FOR 113000 h

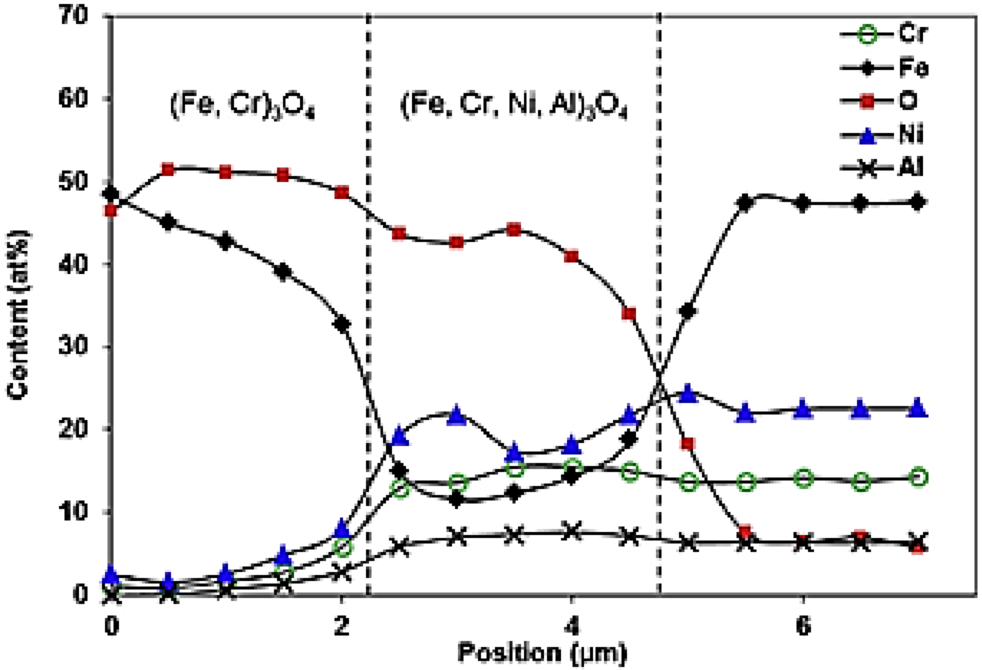
- PHENOMENOLOGICAL MODEL DESCRIBING LIQUID-METAL EMBRITTLEMENT OF STEELS (LME) IN HEAVY-LIQUID METALS DEVELOPED IN PhMI NASU

Scarce literature data:

M. Roy, L. Martinelli, K. Ginestar et al.,
Journal of Nuclear Materials 468 (2016) 153-163

Fe-14Cr-25Ni-3.5Al-2Mn-2Mo-2.5Nb



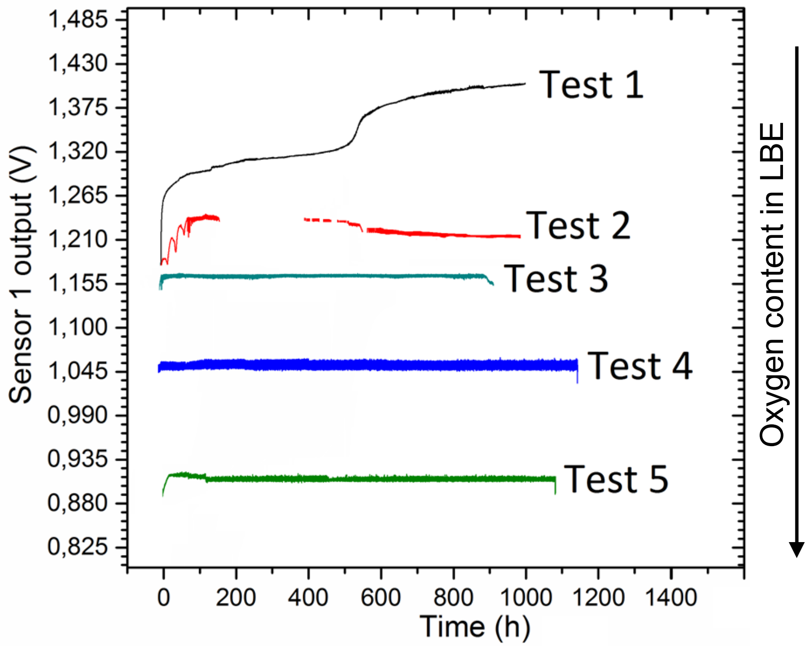


- Static Pb-Bi
- 520°C
- $10^{-9} \leq [O] \leq 5x10^{-4}$
- 1850 h

- The test was carried out mostly at comparable high oxygen concentration in the LBE, i.e. ~10^{-4} wt%
- Bi-layer magnetite scale formed on the AFA alloy shows that there is no substantial gain in using this alloy in comparison with conventional austenitic steels not-alloyed by Al

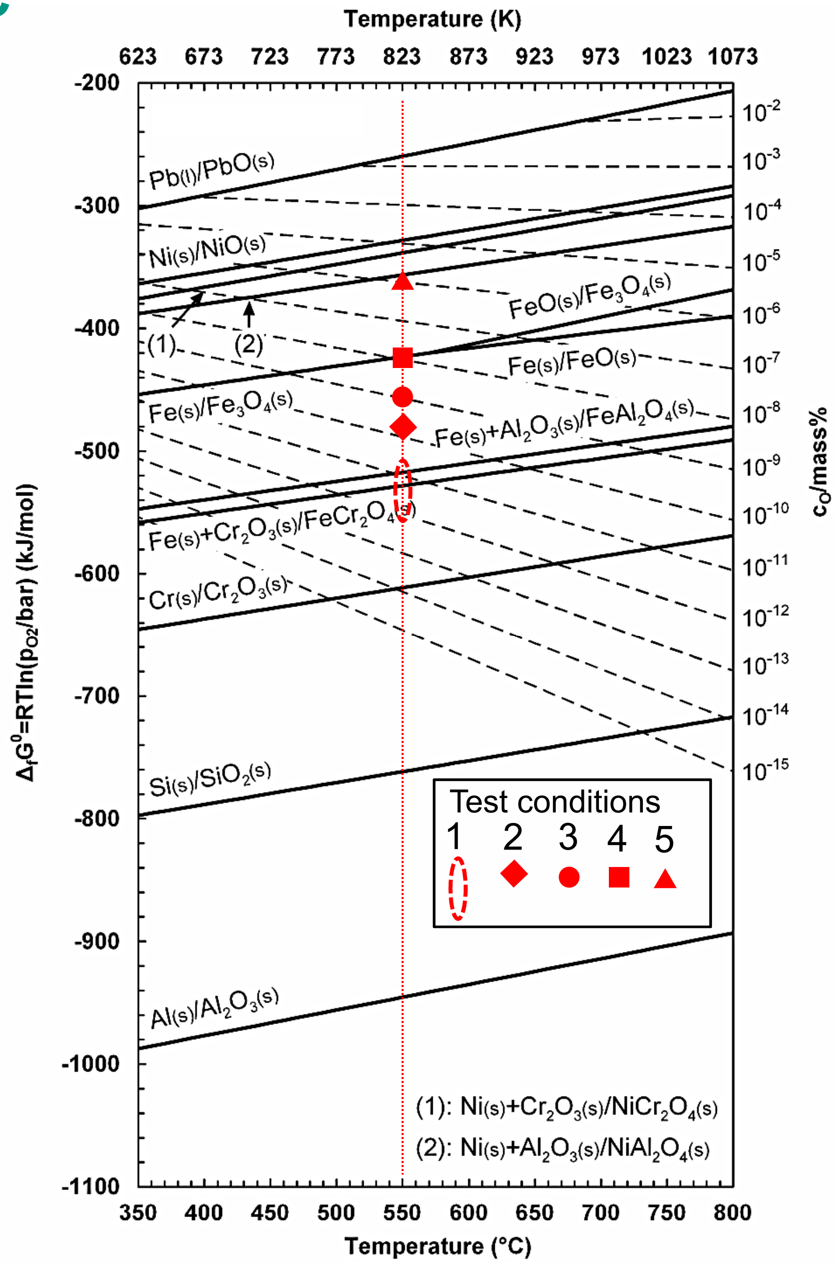
CORROSION TESTS IN Pb-Bi EUTECTIC

- Constant parameters of test:
  - volume of Pb-Bi eutectic (2 kg)
  - ratio of Pb-Bi volume to surface of samples is 25 cm
  - temperature 550°C
  - exposure time ~1000 h
- Varying oxygen concentration in Pb-Bi eutectic
  - Test 1: 10^{-11} -10^{-12} mass%O
  - Test 2: 10^{-10} mass%O
  - Test 3: 10^{-9} mass%O
  - Test 4: 10^{-8} mass%O
  - Test 5: 10^{-6} mass%O



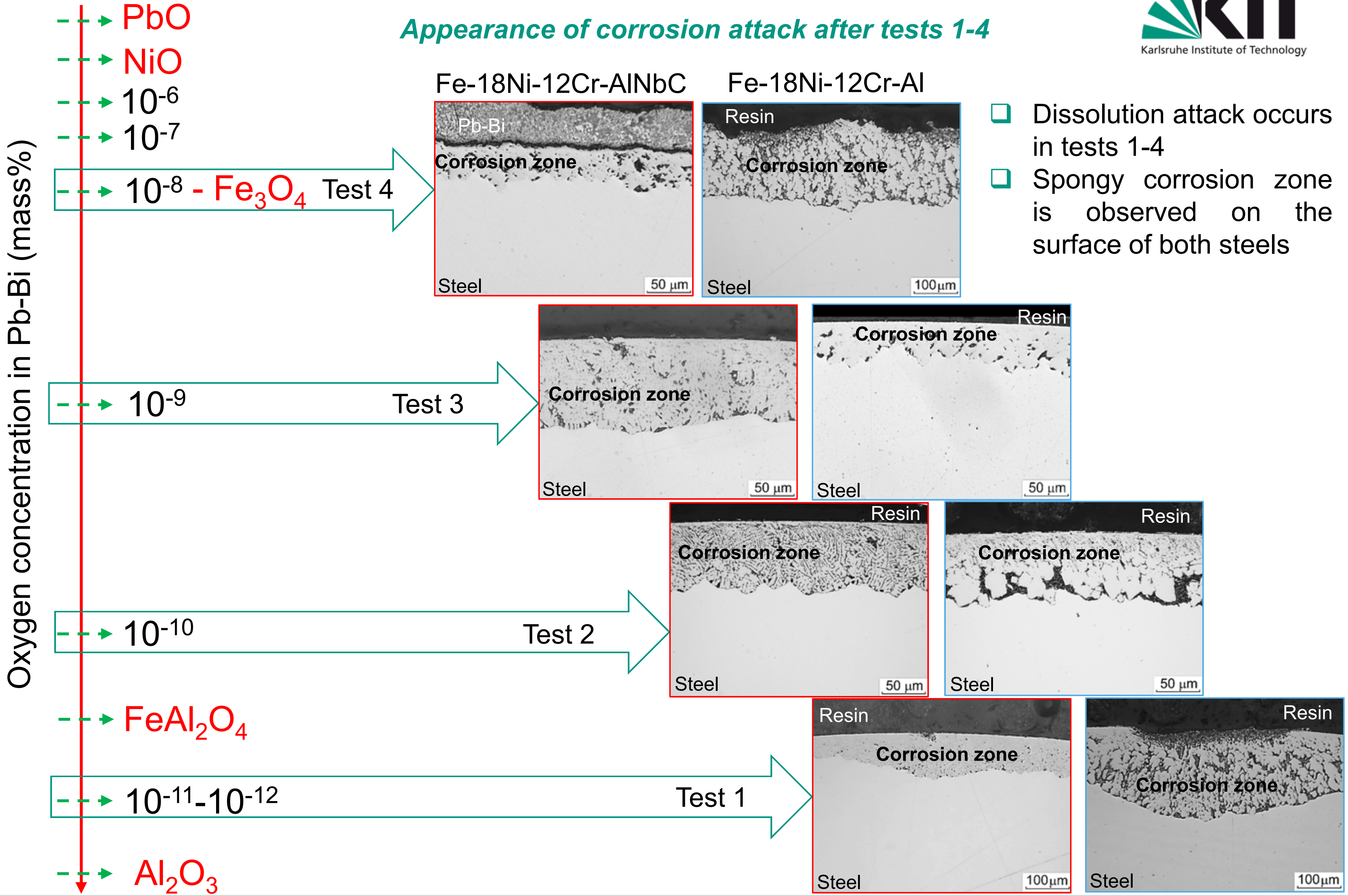
Recalculation of sensor output into the oxygen concentration:

$$log(CO_{Pb-Bi}) = -3.2837 + \frac{6949.8}{T} - 10080\frac{E}{T}$$



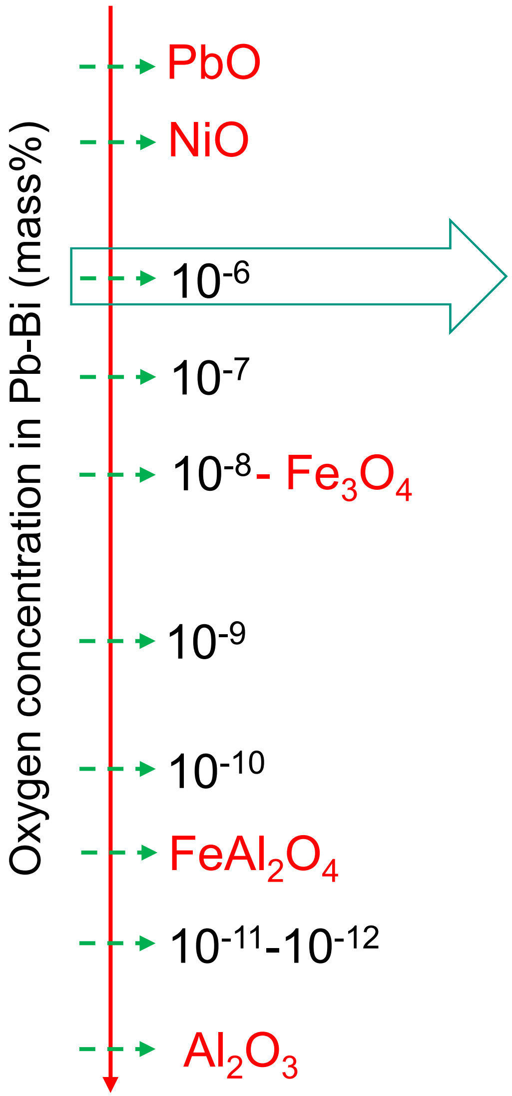
RESULTS OF CORROSION TESTS #1-4

Appearance of corrosion attack after tests 1-4



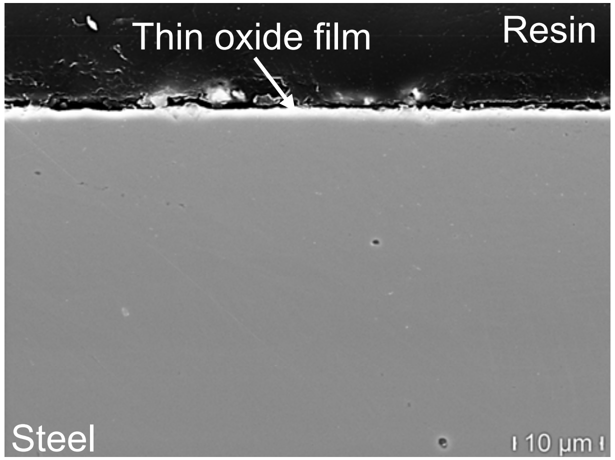
- Dissolution attack occurs in tests 1-4
- Spongy corrosion zone is observed on the surface of both steels

CORROSION TEST 5



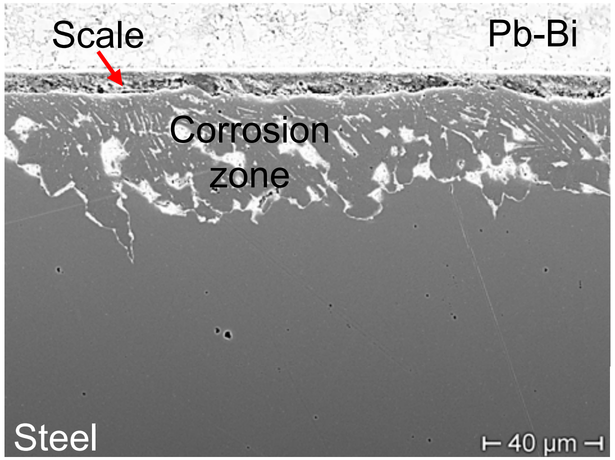
General corrosion appearances on AFA steels

Fe-18Ni-12Cr-AlNbC



Slight oxidation is observed on 80% of surface)

Fe-18Ni-12Cr-Al



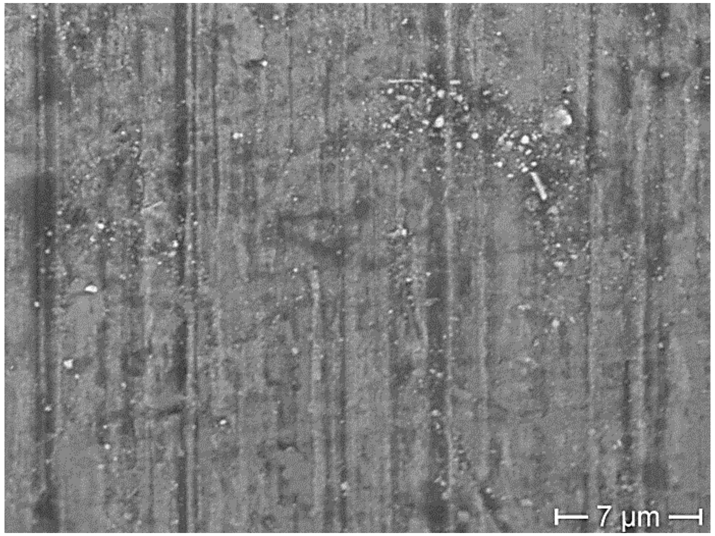
Dissolution + oxidation

- Slight oxidation reflects the general corrosion trend in the case of Fe-18Ni-12Cr-AlNbC steel
- Dissolution attack in combination with oxidation reflects the general corrosion trend on Fe-18Ni-12Cr-Al steel

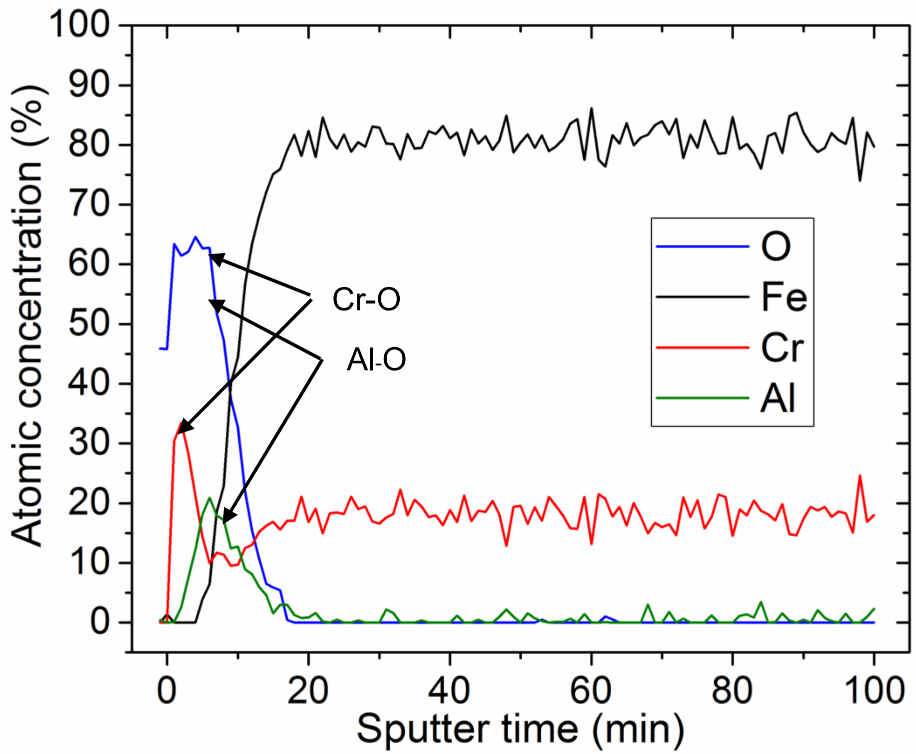
CORROSION TEST 5

Characterization of general corrosion appearance (80%) on Fe-18Ni-12Cr-AlNbC steel

Surface morphology



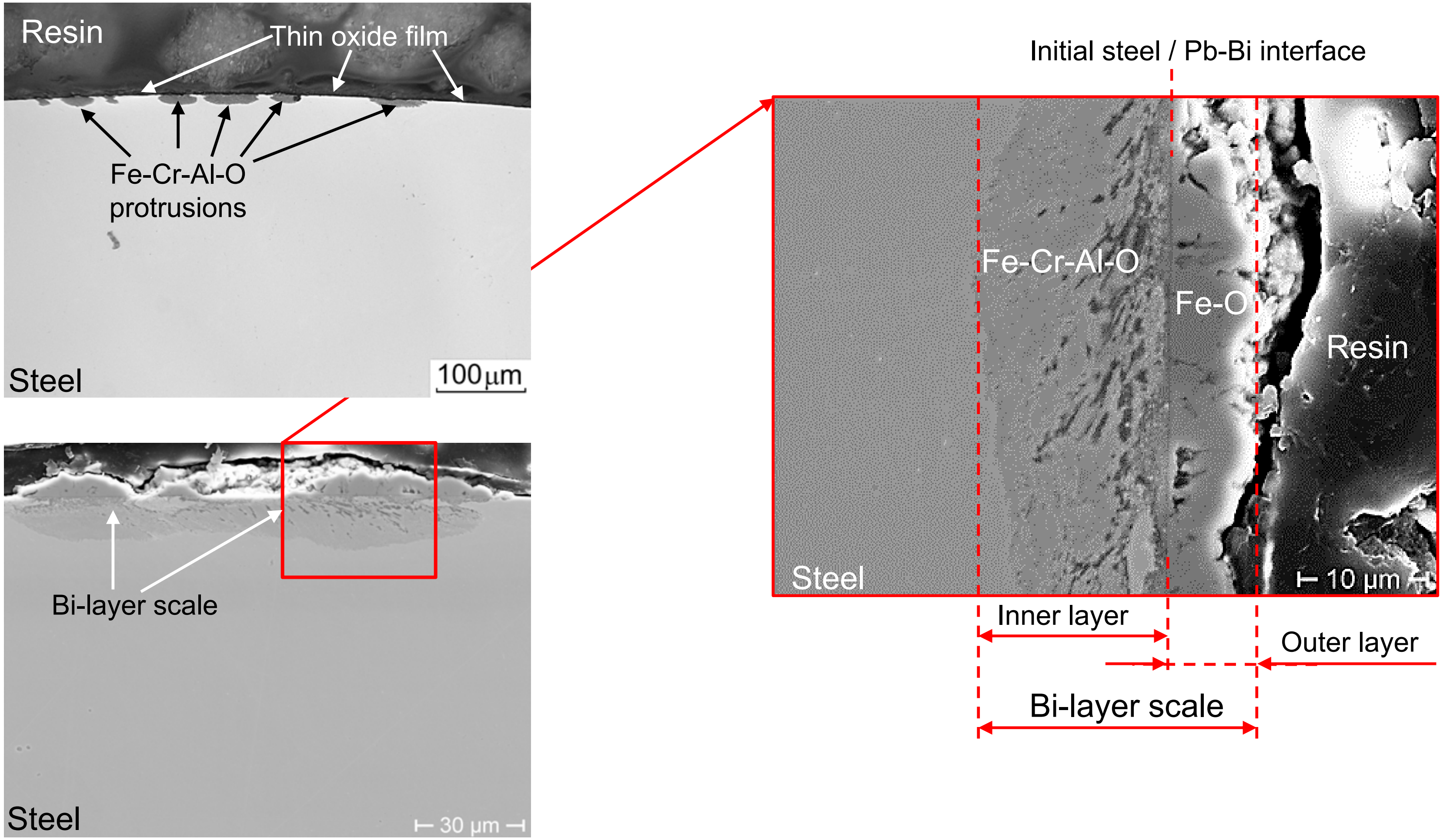
Auger sputter depth profile from surface



- Cr/Al-rich oxide film (on 80% of surface appearance) is formed on steel surface indicating synergetic effect of Cr and Al on the formation of oxide layer

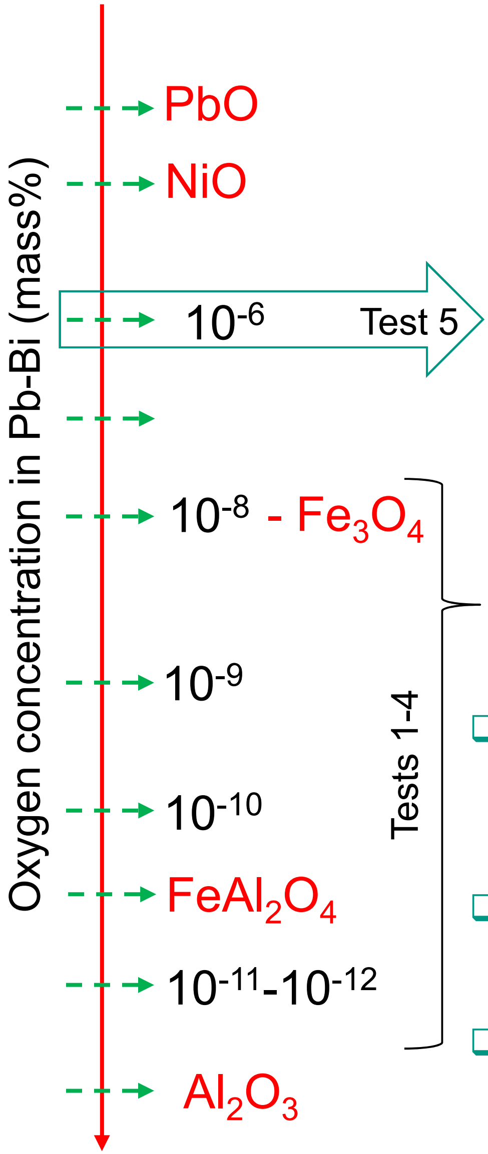
CORROSION TEST 5

Characterization of local corrosion appearances (20%) on Fe-18Ni-12Cr-AlNbC steel

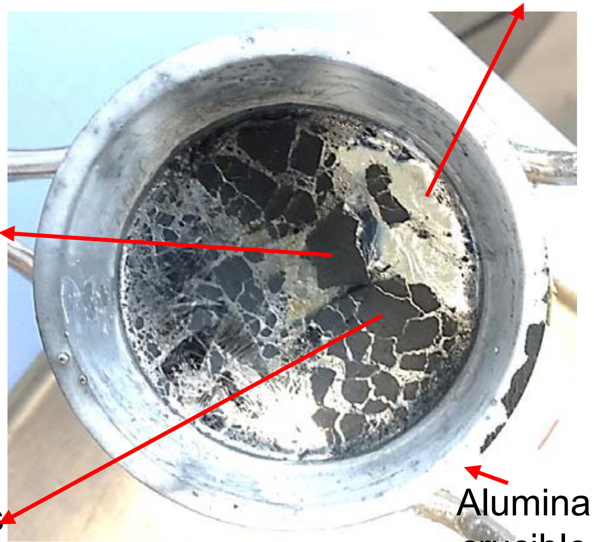


- ❑ Local protrusions of bi-layer magnetite scale or inner Fe-Cr-Al-O spinel are observed
- ❑ Local accelerated oxidation is observed on 20% of surface

CHEMICAL COMPOSITION OF LIQUID METAL AFTER TESTS



Surface of the Pb-Bi bath with floating oxides



- Oxygen from the gas phase reacts with dissolved constituents of steel and forms floating oxides on the surface of liquid metal bath
- Cr-based oxides are forming on the surface of LBE when concentration of oxygen in Pb-Bi is ≤ than necessary for the stability of magnetite
- Fe-based oxides are formed on the surface of LBE when concentration of oxygen in Pb-Bi is > than necessary for the stability of magnetite

Composition of LBE after test 5

	mass%
Al	< 0.00001
Cr	< 0.00001
Fe	< 0.00001
Ni	0.00432 (±0.00001)

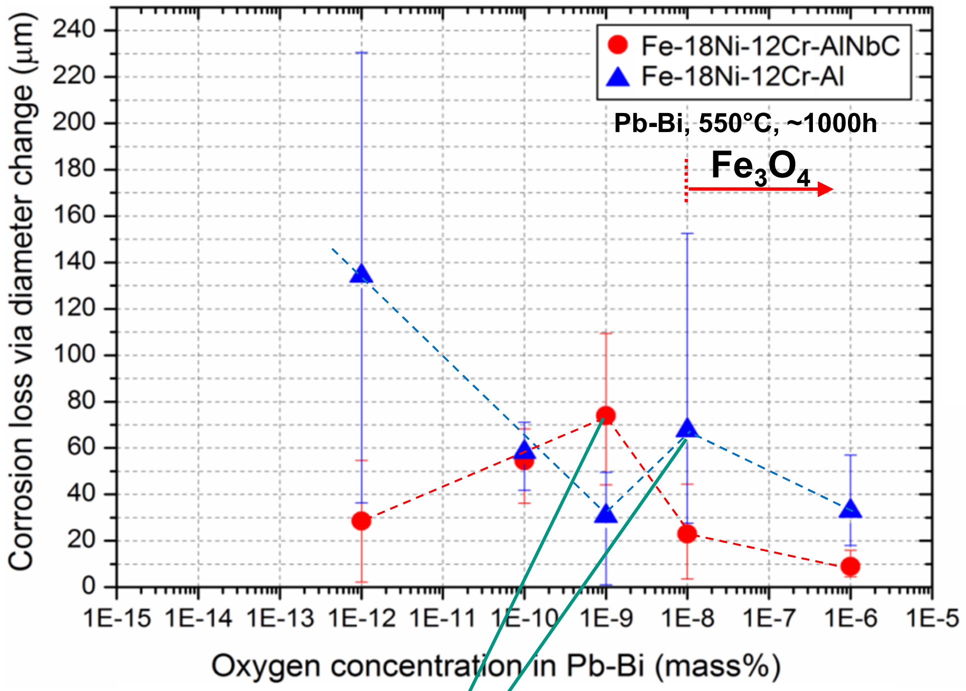
Saturation concentration At 550°C (mass%)	
Al	-
Cr	0.0016
Fe	0.00048
Ni	3.2

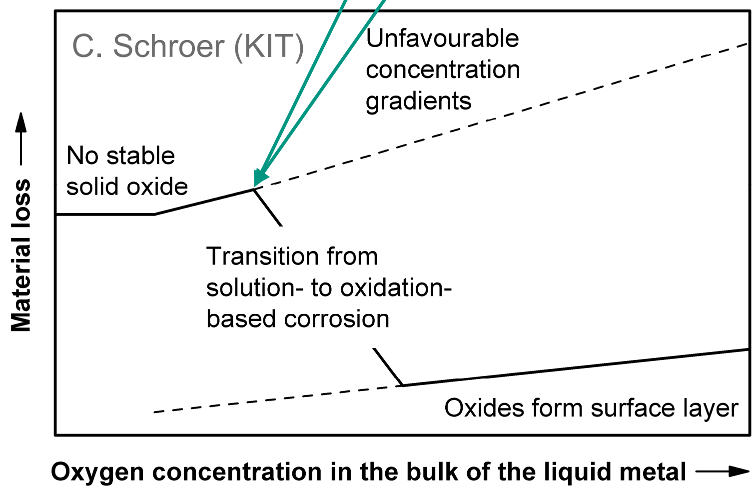
Composition of LBE after test 1

	mass%
Al	< 0.00005
Cr	0.00019 (±0.00002)
Fe	0.00023 (±0.00007)
Ni	0.00230 (±0.00004)

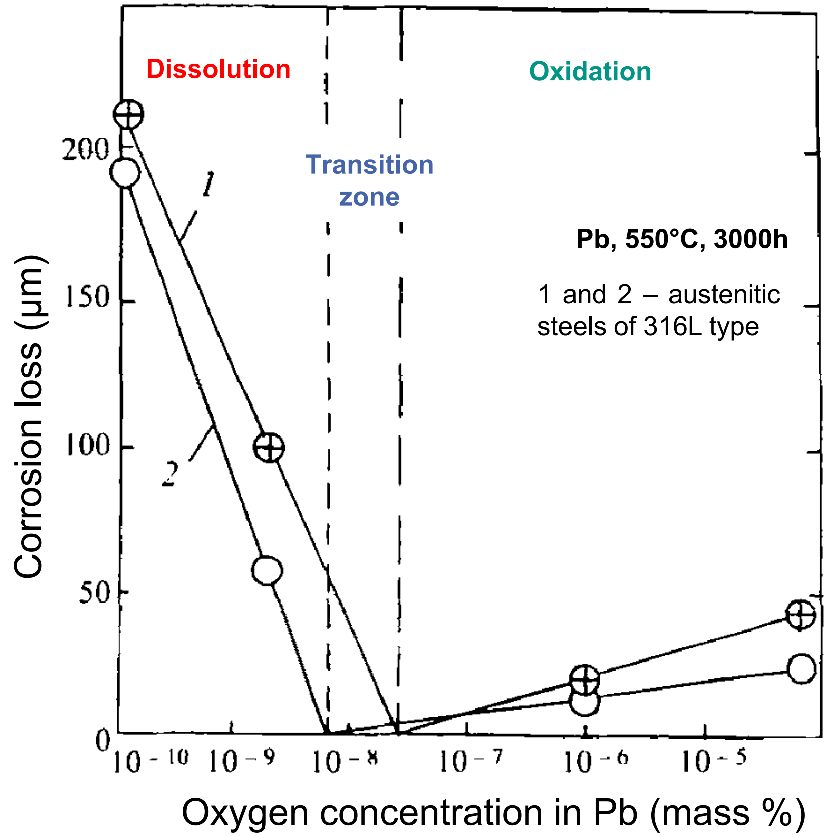
QUANTIFICATION OF CORROSION LOSS

This work





Earlier literature data



I.V. Gorynin et al. Met. Sci. Heat Treat. 41 (9) (1999) 384–388

- Increase in corrosion loss in transition zone ($C_{O[Pb-Bi]} \leq Fe_3O_4$) is observed in comparison with lower oxygen concentrations in LBE

SUMMARY on corrosion of aluminium-alloyed austenitic steels in HLM

Karlsruhe Institute of Technology

- The effect of oxygen concentration in static Pb at 700°C and Pb-Bi eutectic at 550°C on the corrosion behavior of Fe-18Ni-12Cr-2.3Al and Fe-18Ni-12Cr-2.9Al-Nb-C austenitic steels is investigated for about 1000 h
- The oxidation potential of the liquid metal, similar to the conventional austenitic steels not-alloyed by Al, should be higher than required for the thermodynamic stability of magnetite (Fe_3O_4) in order to promote oxidation of AFA steels in Pb and Pb-Bi eutectic
- The more complex alloying in Fe-18Ni-12Cr-2.9Al-Nb-C steel seems favors the formation of more protective oxide film
- Single layer of Al_20_3 is not formed while the multi-layer oxides are detected: Cr/Al-O in Pb-Bi and Fe/Cr/Al-O in Pb
- **Long-term tests under the flowing conditions are necessary to investigate the viability of thin Fe/Cr/Al-based oxide film**

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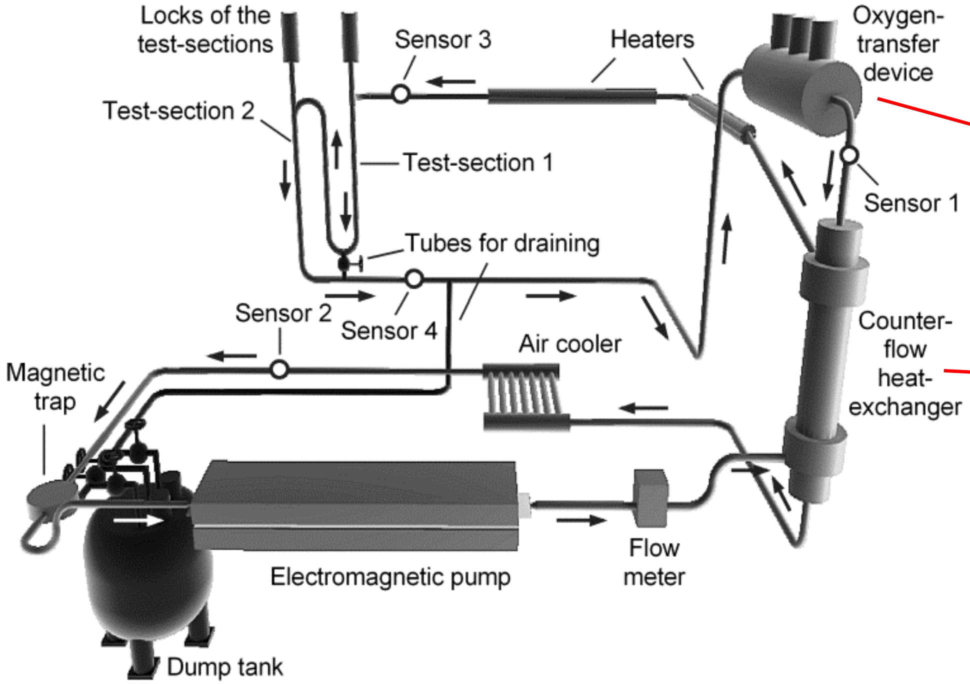
***CORR**osion **I**n **D**ynamic lead **A**lloys - **CORRIDA** loop*



The CORRIDA facility – a forced-convection loop made of austenitic stainless steel (1.4571) designed to expose material (steel) specimens to flowing (2 m/s) Pb-Bi eutectic (~1000 kg) <u>with controlled oxygen concentration</u>.

Operating history of the CORRIDA loop

Carsten Schroer (KIT), ICONE26

Karlsruhe Institute of Technology

Commissioning in Feb 2003.

Start of operation at 550 °C/ 10^{-6} % oxygen in Jul 2003.
Oxygen transfer with Ar–H_2–H_2O.

Manual air addition to Ar–H_2–H_2O. ☺

Optimization.

Dry gas.

Optimized manual air addition to humidified Ar. ☺
Tube failure at the inlet of the second test-section after total operation for 29,000 h. ☹

450 °C/10^{-6} % oxygen.

Return to 550 °C/ 10^{-6} % oxygen.
Automated air addition to Ar or Ar–H_2. ☺
Decreasing mass flow and mobile oxide deposits. ☹

☺ Automation of hydrogen addition.
550 °C/ 10^{-7} % oxygen.
☹ Tube failure at the bottom end of the first test-section after total operation for 66,000 h.

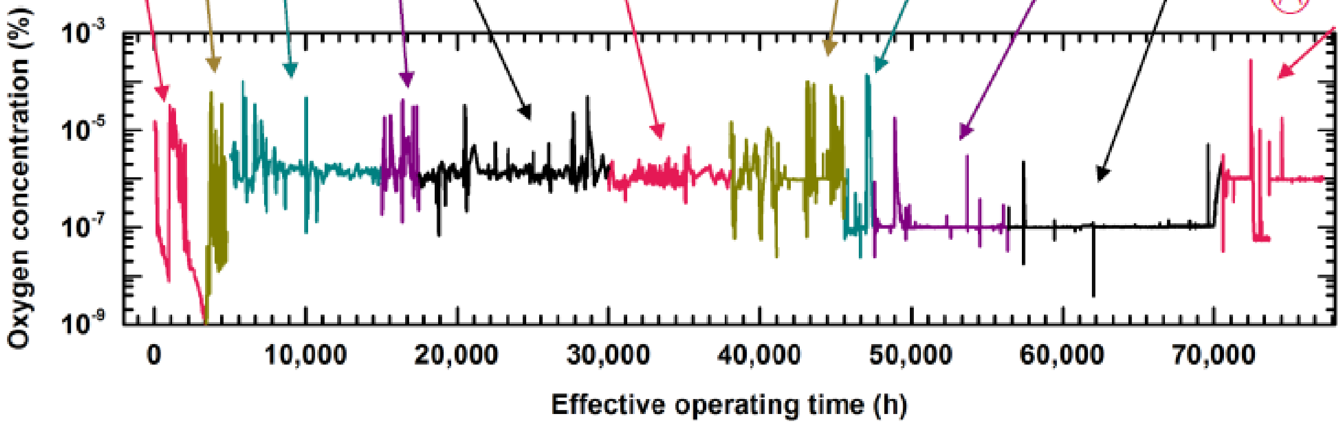
450 °C/ 10^{-7} % oxygen.
☹ Tube failure close to the weldment of the once replaced inlet piece after total operation for 47,500 (76,500) h.

400 °C/ 10^{-7} % oxygen.

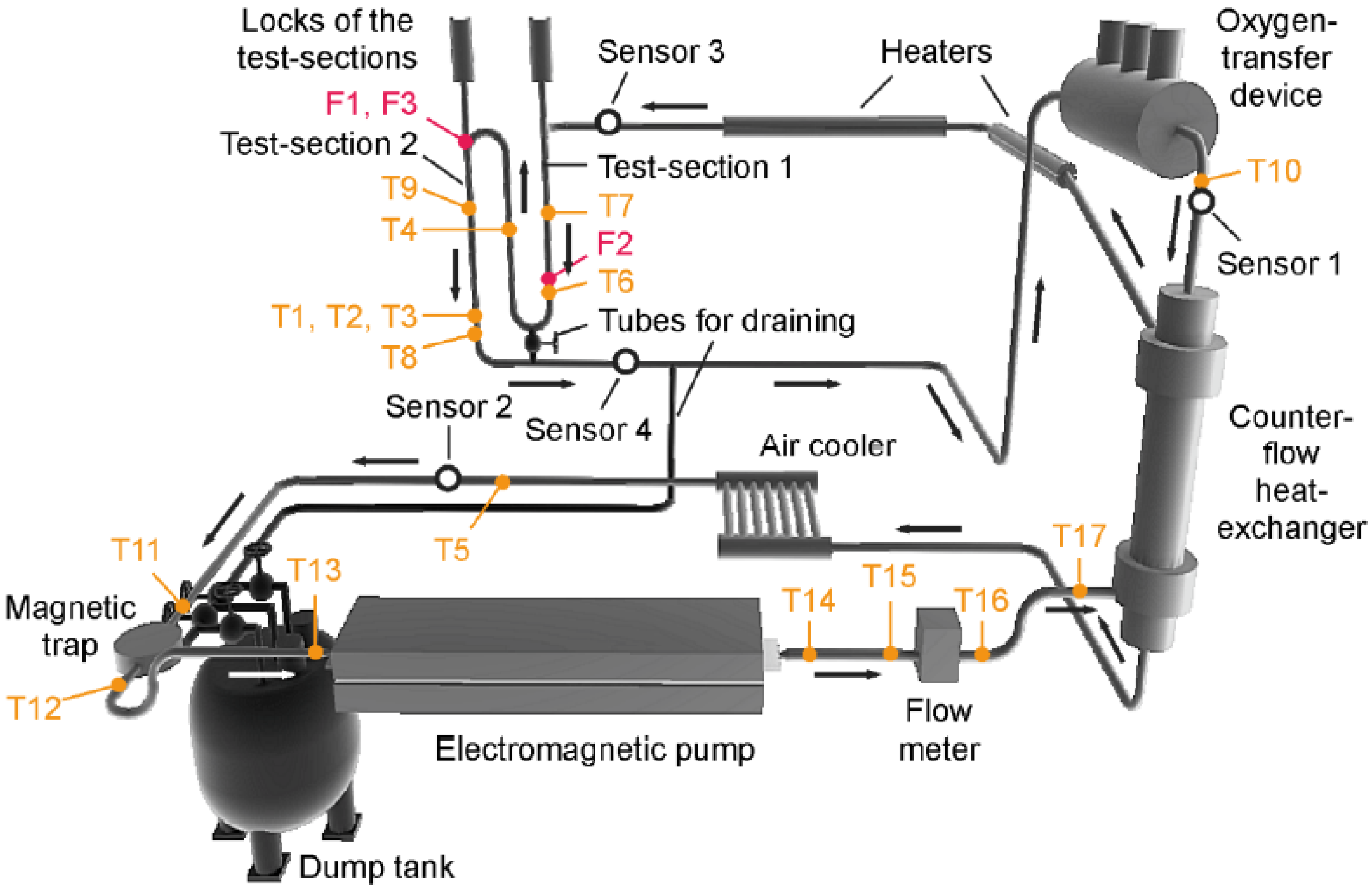
500 °C/ 10^{-6} % oxygen.
☹ Problems with mass flow return.

Total operation for 100,000 h in Feb 2016.
Effective operation for 75,000 h in May 2017. ☺

Plug in the cold leg after total operation for 113,000 h (Dec 2017): Loop cannot be refilled after LBE dump. ☹

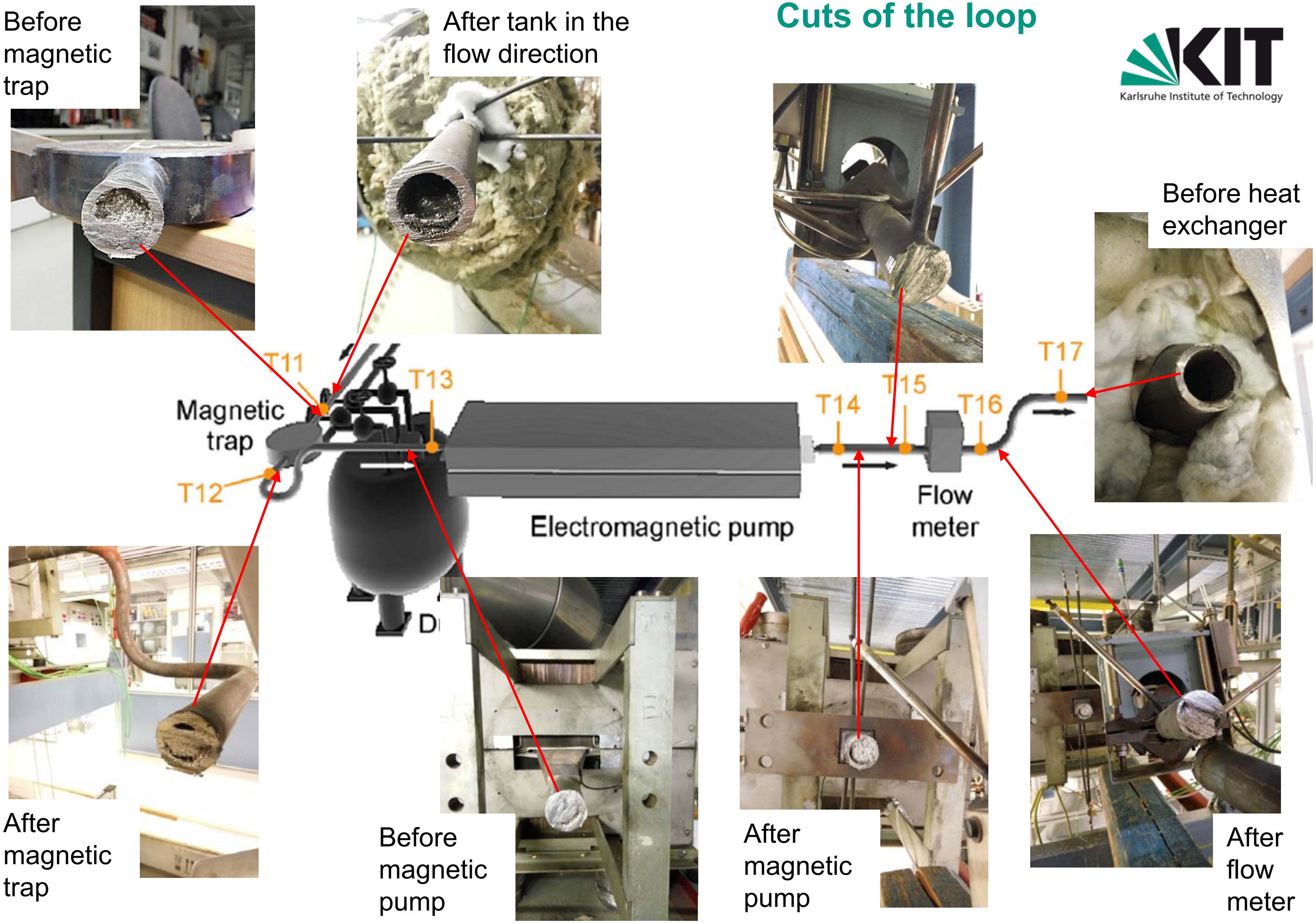


Localization of possible plugging areas in the loop

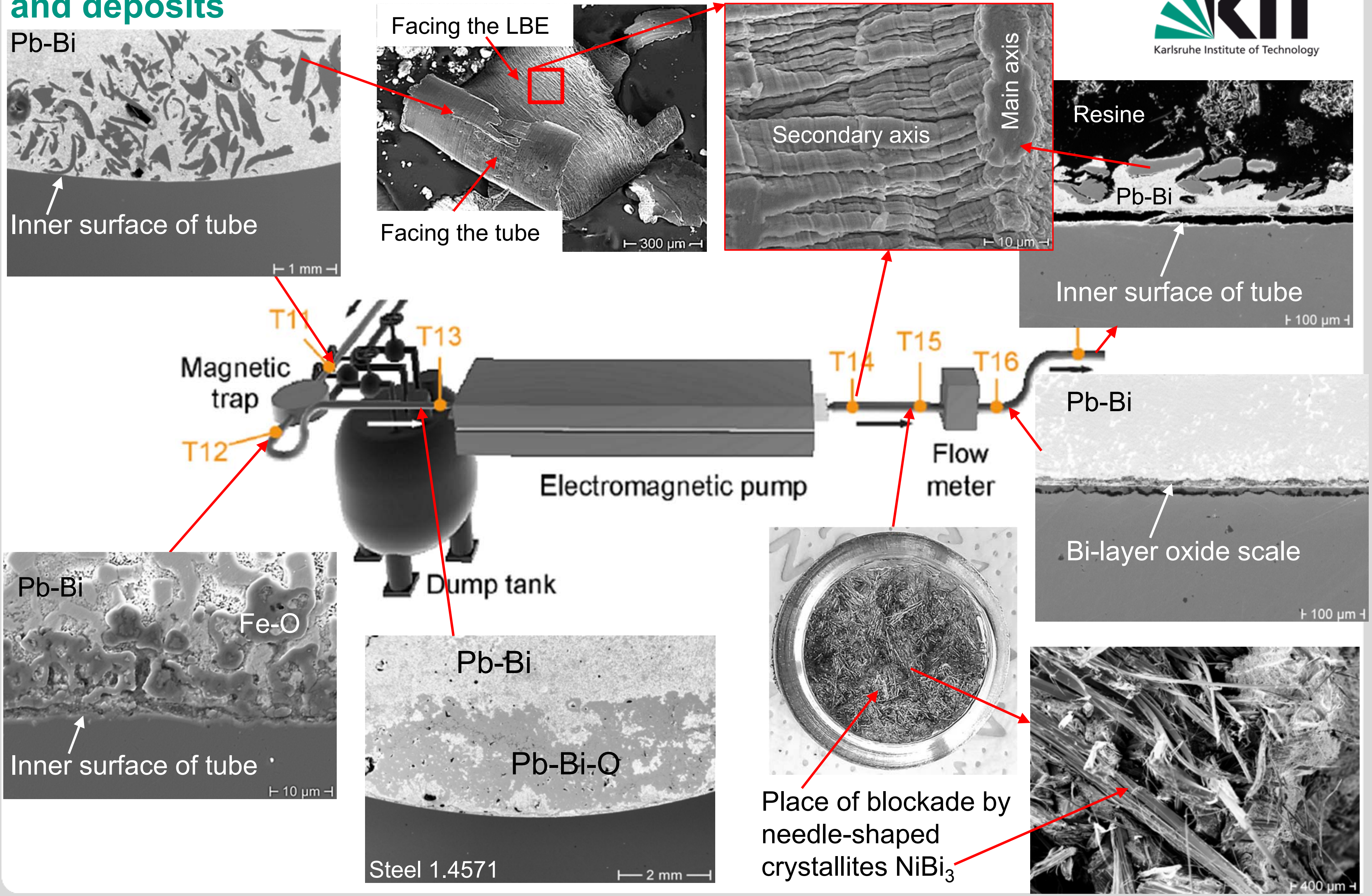


- According to the output of the thermocouples the solidified Pb-Bi is located among thermocouples T11 and T16.

Cuts of the loop



Precipitates and deposits



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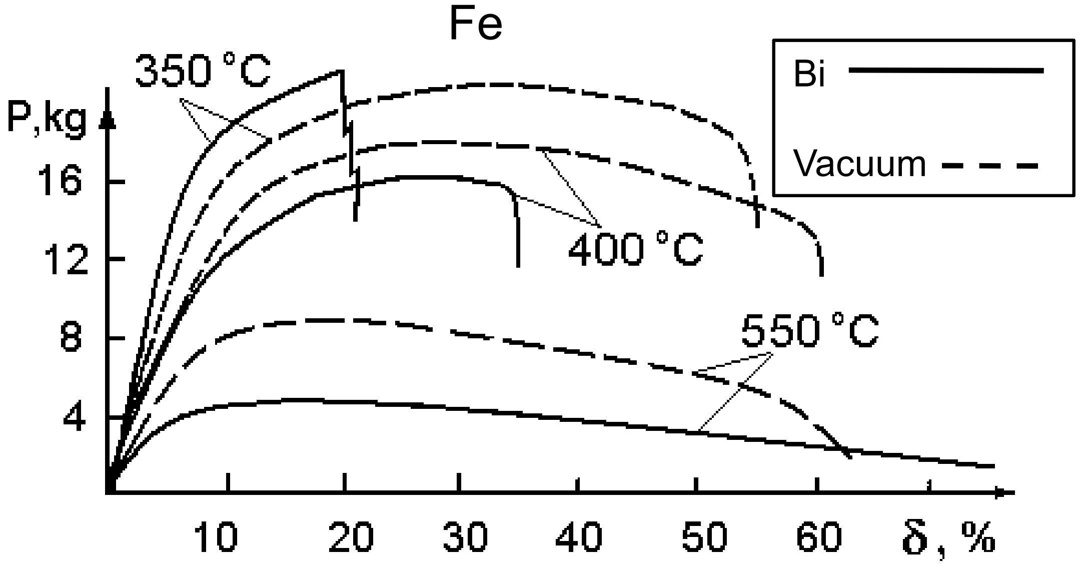
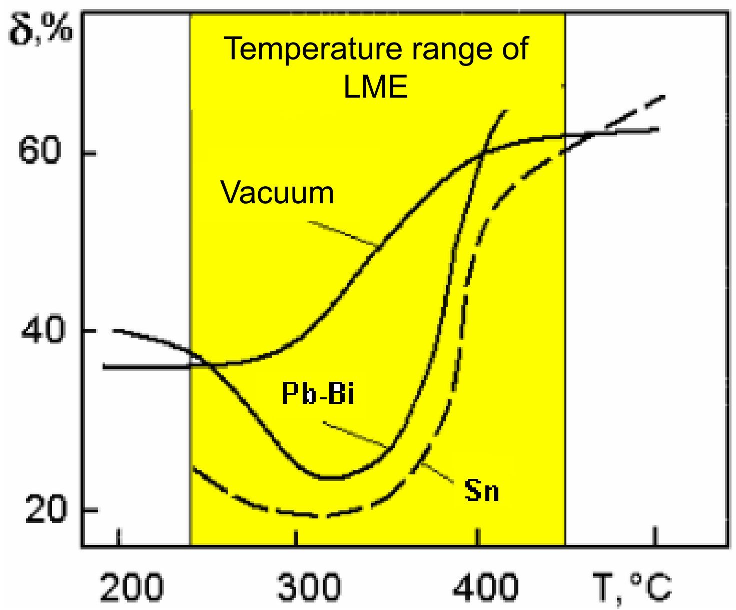
LIQUID-METAL EMBRITTLEMENT (LME) OF STEELS IN HEAVY-LIQUID METALS (HLM)

Karlsruhe Institute of Technology



*Olha Yeliseyeva, PhMI NASU**

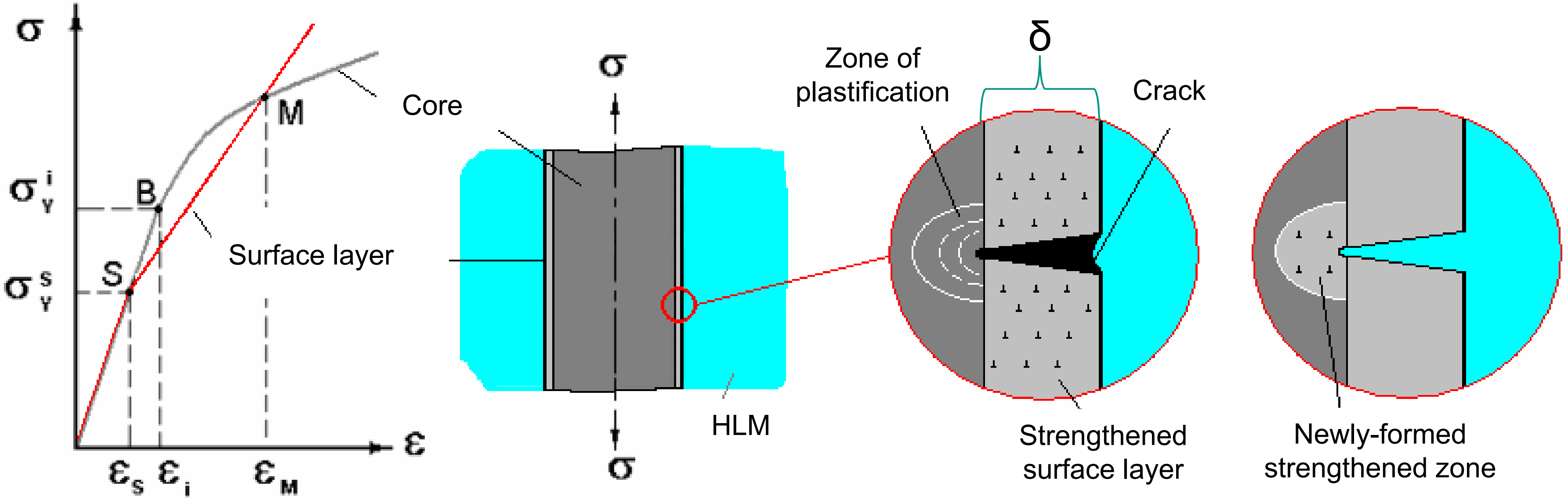


- Liquid metal embrittlement (LME) is a phenomenon which can be defined as the brittle fracture (or loss of ductility) of a usually ductile material in presence of heavy-liquid metals (HLM) when external stress is applied.
- Wetting of the material surface by HLM is an important prerequisite for LME to occur.
- LME occurs in the specific temperature range and deformation rate.
- F/M steels suffers from LME while austenitic steels vice-versa show plastification.

** PhMI NASU - Physical–Mechanical Institute of National Academy of Science of Ukraine, 5, Naukova Street, Lviv 79601, Ukraine*
http://www.ipm.lviv.ua/new/eng/index.php

PHENOMENOLOGICAL MODEL DESCRIBING LIQUID-METAL EMBRITTLEMENT OF STEELS IN HEAVY-LIQUID METALS

V. Popovich, I. Dmuchovska, PhMI NASU



*Olha Yeliseyeva, PhMI NASU**

1. Facilitating the plastic flow of a thin surface layer δ of solid metal due to the reduction of surface energy by absorbed liquid metal and as a result facilitating the movement of dislocations under the simultaneous action of a melt and tensile stress (red line)
2. Active plastic deformation of surface layer intensifies in turn the deformation strengthening, in comparison with core, that facilitates origin and distribution of surface cracks on the depth δ.
3. After passing the strengthened layer, the cracks facing the ductile material and arrested.
4. When liquid metal percolate the crack and facing ductile material the process repeats.

Materials Science, Volume 18, Issue 6, 1982, Pages 461-467. https://link.springer.com/article/10.1007/BF00729424

Thank you for attention !!!

Karlsruhe Institute of Technology