KINETICS & MECHANISM OF CRACK INITIATION OF LME

2nd level testing - the EAC understanding

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CVŘ Centrum výzkumu Řež

GEMMA Generation IV Materials Maturity

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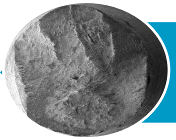
R4S RESEARCH FOR SUSEN

Skupina ÚJV TECHNOLOGIE I INOVACE I LIDÉ

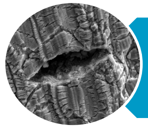
Outline

Motivation

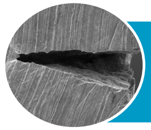
Introduction



Results I - Sensitivity to LME/EAC cracking in HLM



Results II - Conditions for LME/EAC cracking in HLM: Initiation



Results III - Conditions for LME/EAC cracking in HLM: Kinetics

Conclusions

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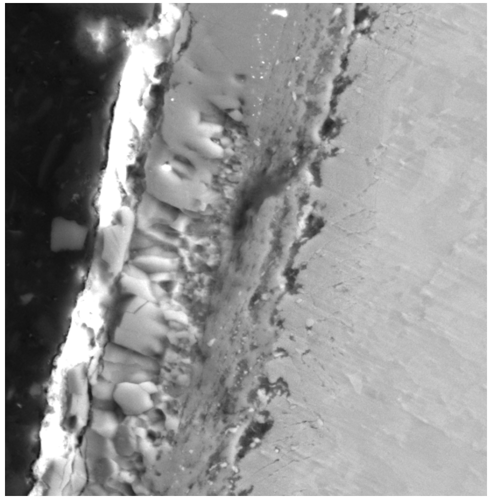
Motivation



Material degradation in HLM:
→ **Dissolution Corrosion**
If stress is applied
→ **Environmentally Assisted Cracking** (EAC)

Present selection of candidate structural materials for internal components of LFR:

15-15Ti: good corrosion, but local effects; resistant to EAC; no DBTT, low swelling

T91: small corrosion, **sensitivity to EAC**, very good behavior under irradiation (no swelling, little DBTT)

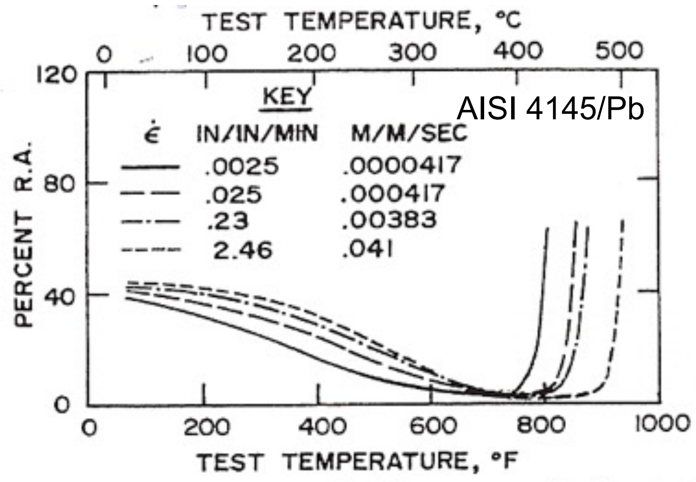
The phenomenon called **Liquid Metal Embrittlement** (LME) is had been studied for 40 years.

Performance of T91 in HLM had been studied in several EC projects – TECLA, EUROTRANS, … MATTER…etc..

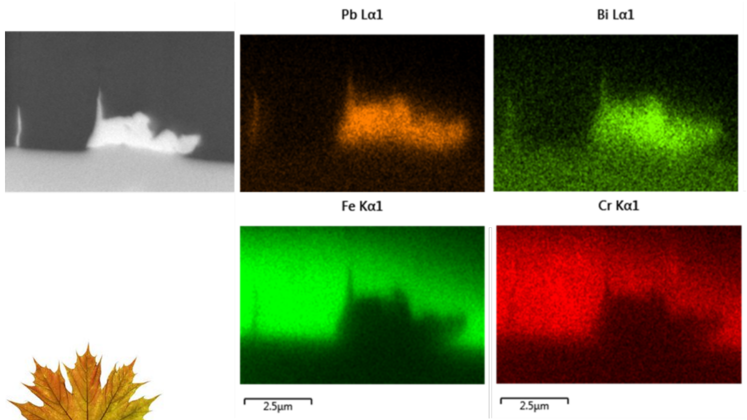
Motivation: Cracking in HLM

Cracking in HLM
- Occurrence close to T_M (LBE: 200-350 °C; Pb: 350-400 °C)
- Crack growth >> Diffusion
- Adsorption based models (AIDE, etc.)

History: **Ductility trough**
Ambiguous explanation:
Embrittlement (LME) or EAC?



Johnson, K.L. in Environmental degradation of engineering materials, 1977, Blacksburg, Virginia
Ex: AISI 4145/Pb



The LME is likely EAC, **LME/EAC**.
Pb, Bi atoms do not penetrate into bulk at 300-400 °C
Confirmed by WDS SEM and ToF-SIMS (Serre, I.P., AppSurfSci 2019)

Introduction

The phenomenon of cracking in HLM had been studied for many years, but the conditions for the crack initiation were not determined yet.

To fully verify material performance in HLM under stress

Two-level experimenting is needed

1st Level: Sensitivity to cracking to be tested

2nd Level: More understanding → characteristics of the degradation to be investigated

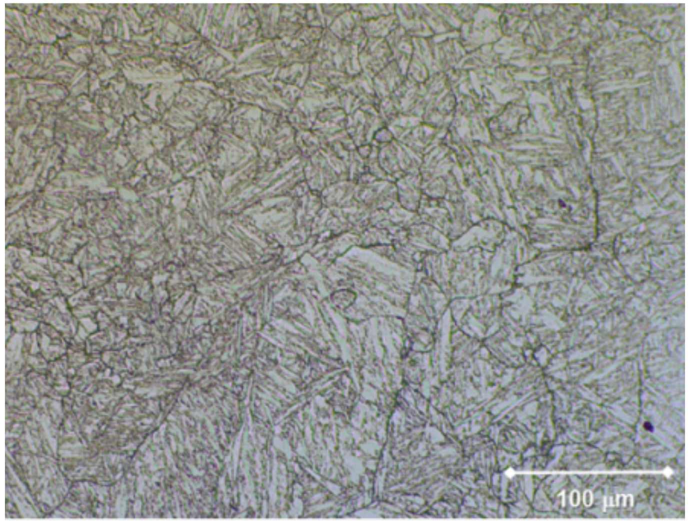
↓

The paper aims to summarize and to sort new and previous data of the two candidate materials.

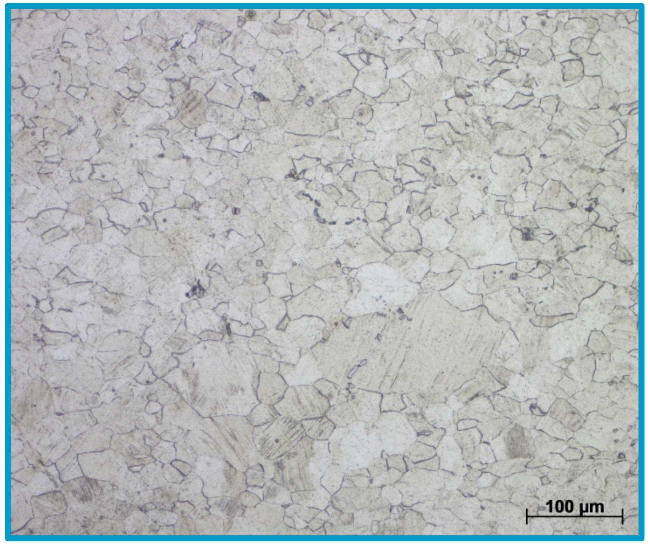
Materials

T91 (Grade 91 Class 2/S50460): Ferritic-martensitic steel - Industeel, ArcelorMittal group - the EUROTRANS/DEMETRA

- 8.895%Cr, 0.889Mo, 0.401Mn, 0.235Si, 0.121Ni, 0.202V, 0.080Cu, 0.048N, 0.019P, 0.010Al, 0.102C, bal. Fe



15-15Ti (1.4970)



Austenitic Ti-stabilized stainless steel

- 15.95%Cr, 15.40Ni, 1.20Mo, 1.49Mn, 0.52Si, 0.44Ti, 0.026Cu, 0.036V, 0.023Al, 0.1C, bal. Fe

- a cylindrical billet of 12cm, after solution annealing.

- non-uniform grain size, from 19 to 67 µm.

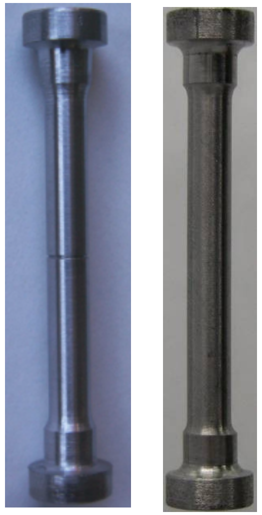
1st Level testing – Sensitivity to LME/EAC cracking in HLM

Experimental

Specimens

Smooth round bar (Ø4 × 20 mm)

Notched round bar (Ø4 × 20 mm)



Test technique

Slow Strain Rate (SSRT) test

- Strain rates
  - R0, 1×10^{-2} 1/s
  - R1, 1×10^{-4} 1/s
  - R2, 1×10^{-6} 1/s

Environment

HLM:

Liquid Lead-Bismuth Eutectic (LBE)

T: 300, 500 ºC

O ≈ 4-100 $\times10^{-8}$ wt. %

Liquid Lead (Pb)

T: “300”, 350, 400, 450 ºC

O ≈ 1-10 $\times10^{-7}$ wt. %

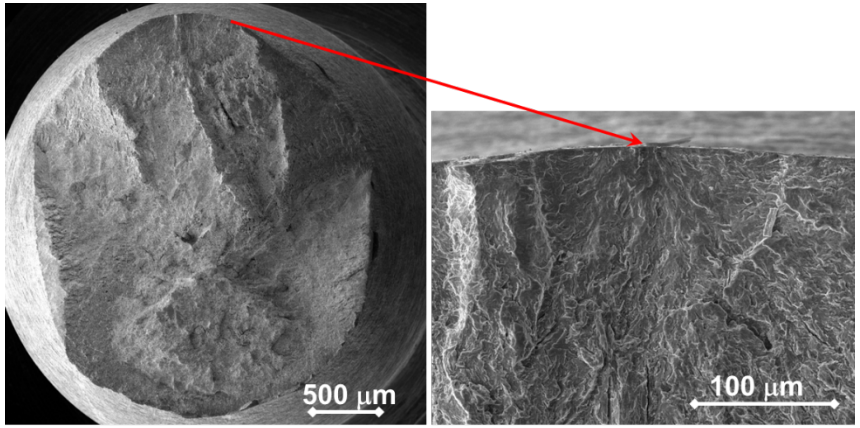
Post-test evaluation

Microscopy observation: SEM + EDS,

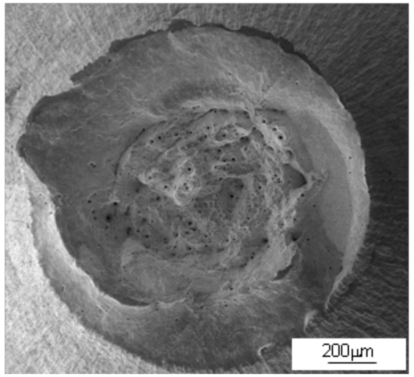
Chemically cleaning in a solution of H_2O_2, CH_3COOH and CH_3CH_2OH (1:1:1)

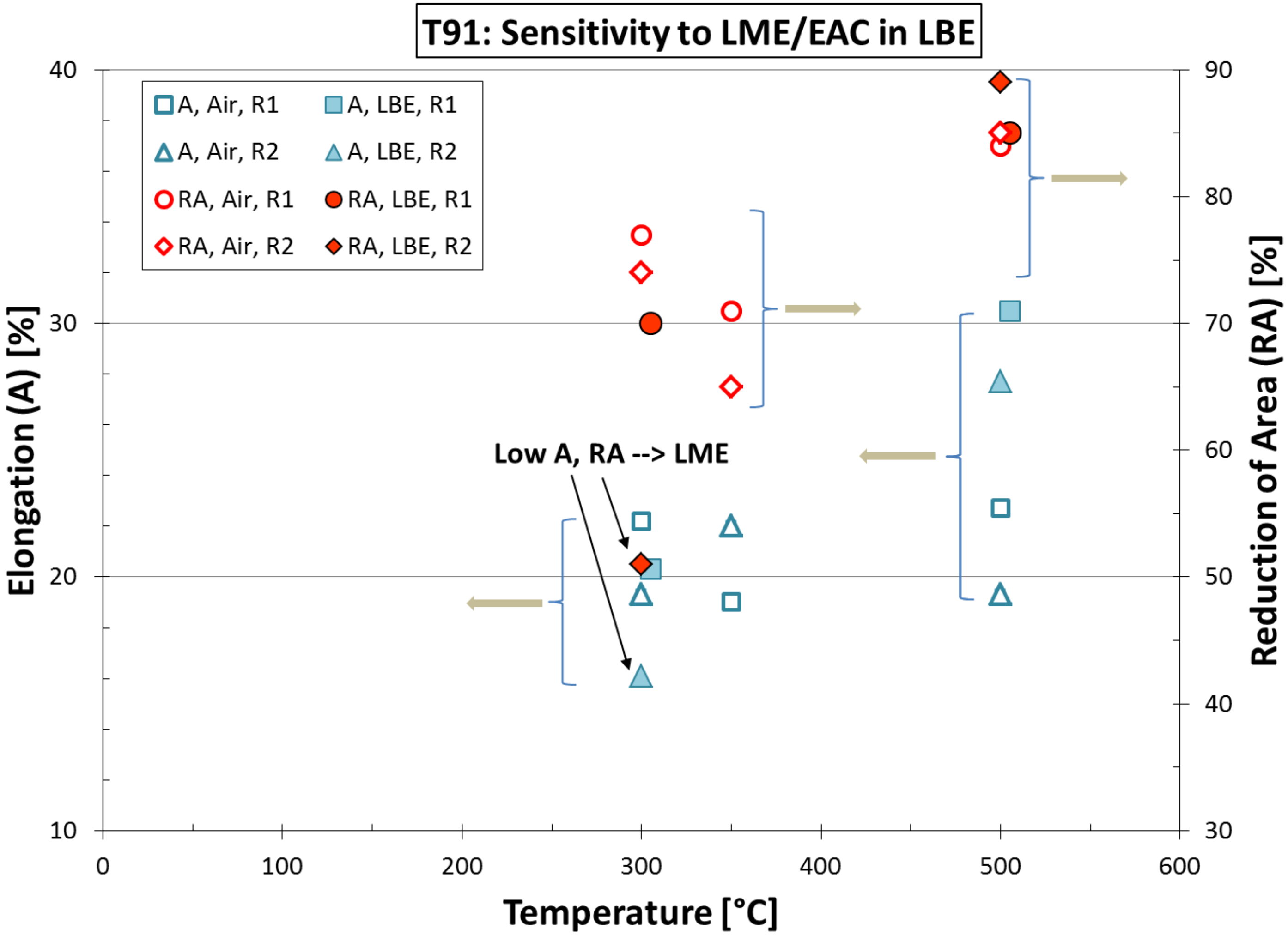
T91 & liquid LBE

300 °C



500 °C

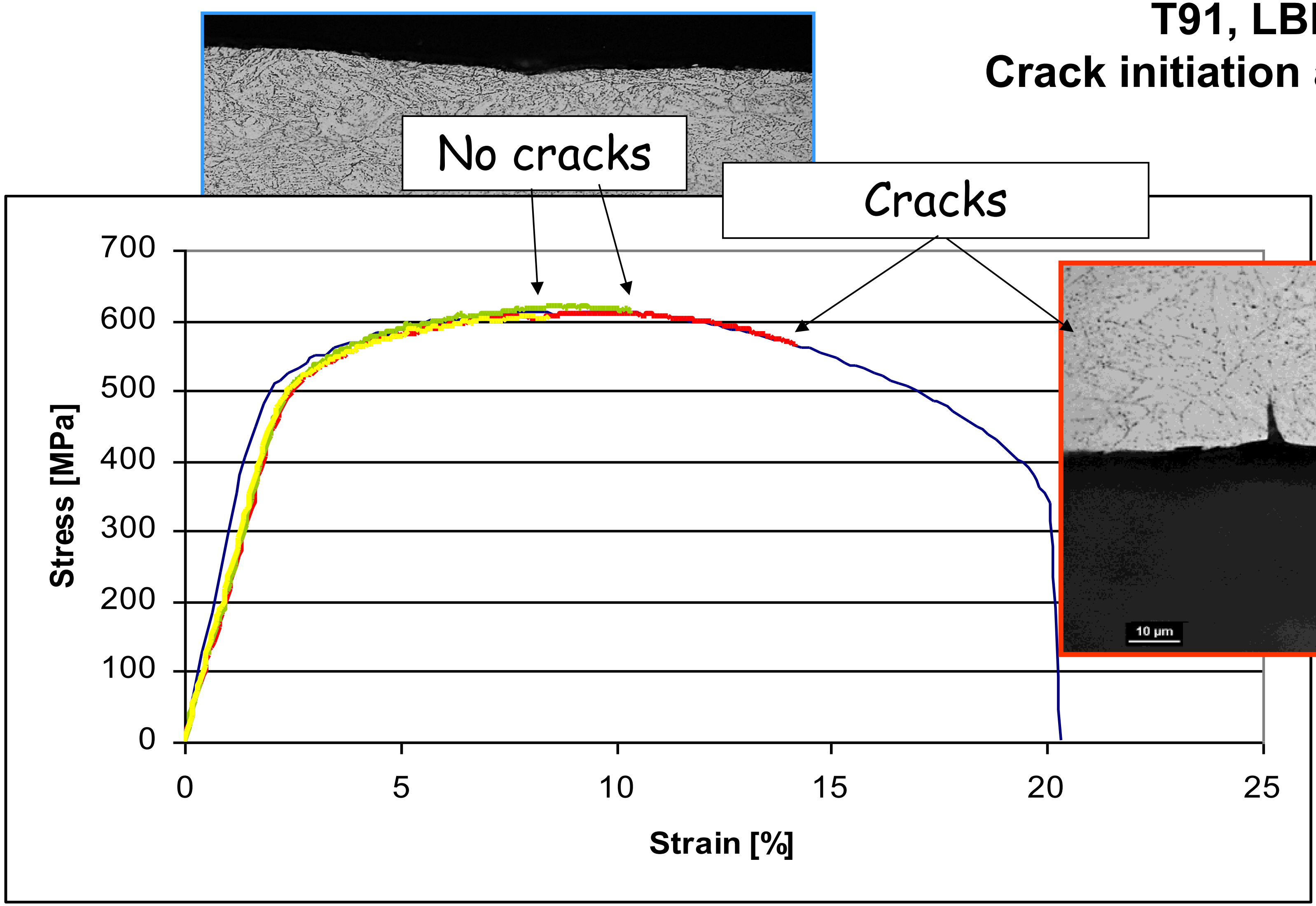


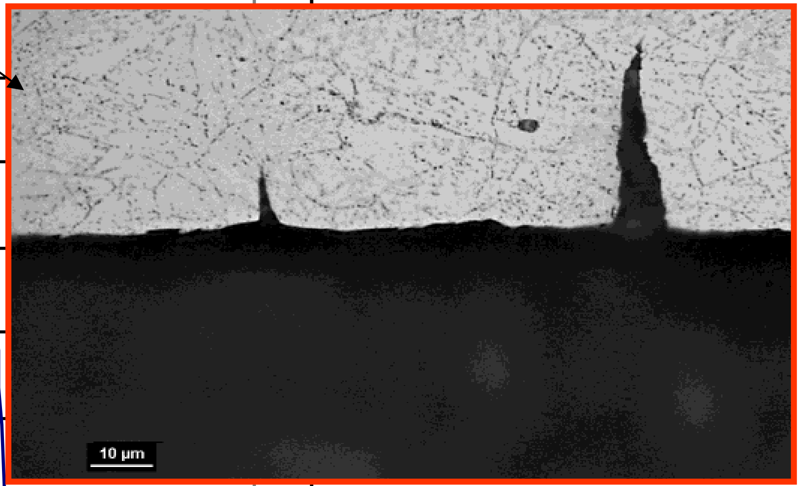


Di Gabriele, F.; Doubkova, A.; Hojna, A. Investigation of the sensitivity to EAC of steel T91 in contact with liquid LBE, J. Nucl. Mater. 376 (2008) 307–311

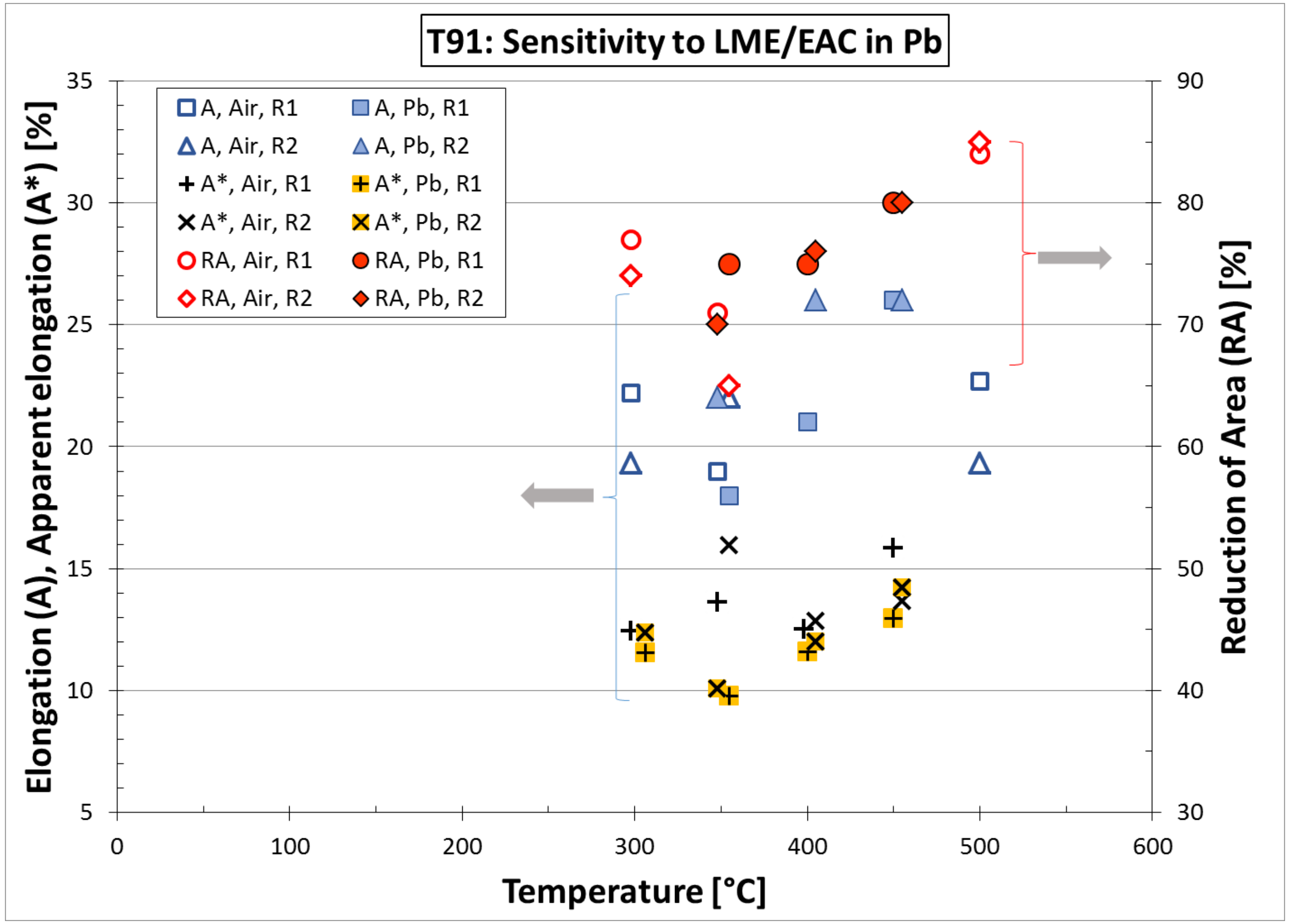
Special observation: Interrupted SSRT

T91, LBE & 300 °C:
Crack initiation at maximum stress



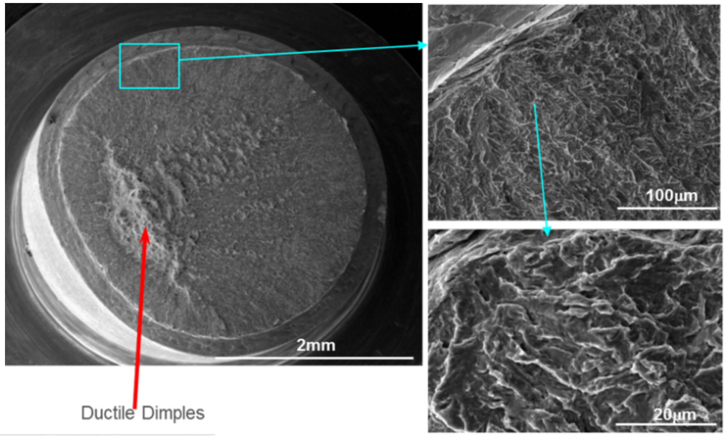


T91 & liquid Pb

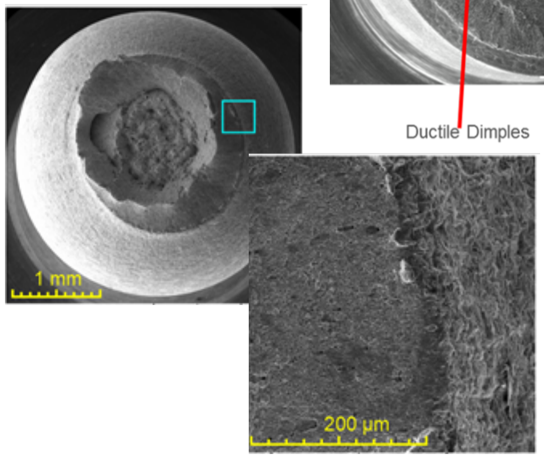


350 °C

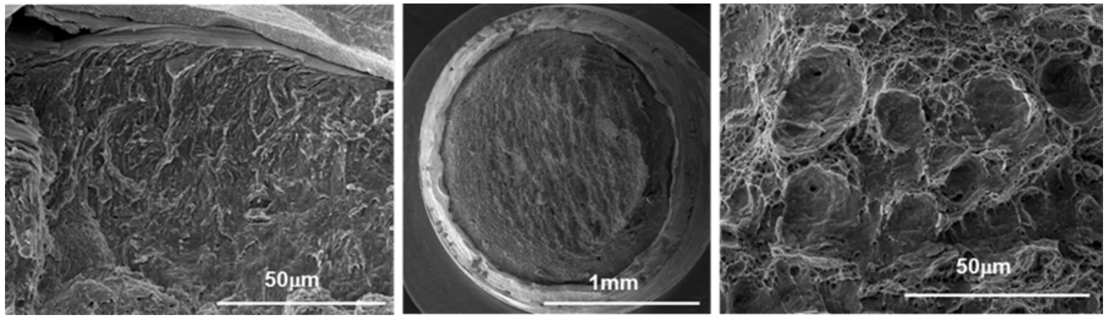
Notched



Smooth



300 °C Notched



Hojna, A.; Di Gabriele, F.; Klecka, J.; Burda, J. J. Nucl. Mater. 466 (2015) 292–301.
Di Gabriele, F.; Hojná, A.; Chocholousek, M.; Klecka, J. Metals 7 (2017) 342-356.

2nd Level testing – Conditions for LME/EAC cracking in HLM: Initiation

Experimental

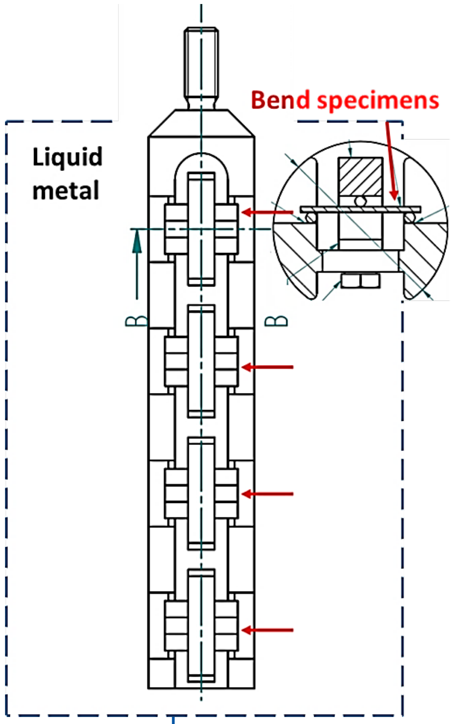
Specimens

Flat coupons (1 × 5 × 22 mm)

Flat tapered platelet (3 × 4-6.4 × 20 mm)

Test technique

- Three-Point Bend Exposure Test - coupons
  - Pre-stressed to 80-110%YS
  - Exposed for 500, 1000 and 2000 hours
- Constant Extension Rate (CERT)
  - Displacement & strain rates
    - R0, 2×10^{-4} m/s $\approx 1\times10^{-2}$ 1/s
    - R1, 2×10^{-6} m/s $\approx 1\times10^{-4}$ 1/s
    - R2, 2×10^{-8} m/s $\approx 1\times10^{-6}$ 1/s



Environment

HLM:

Liquid Lead-Bismuth Eutectic (LBE)

T: 350 ºC

O ≈ 4-100 $\times10^{-8}$ wt. %

Liquid Lead (Pb)

T: 350, 400 ºC

O ≈ 1-10 $\times10^{-7}$ wt. %

Post-test evaluation

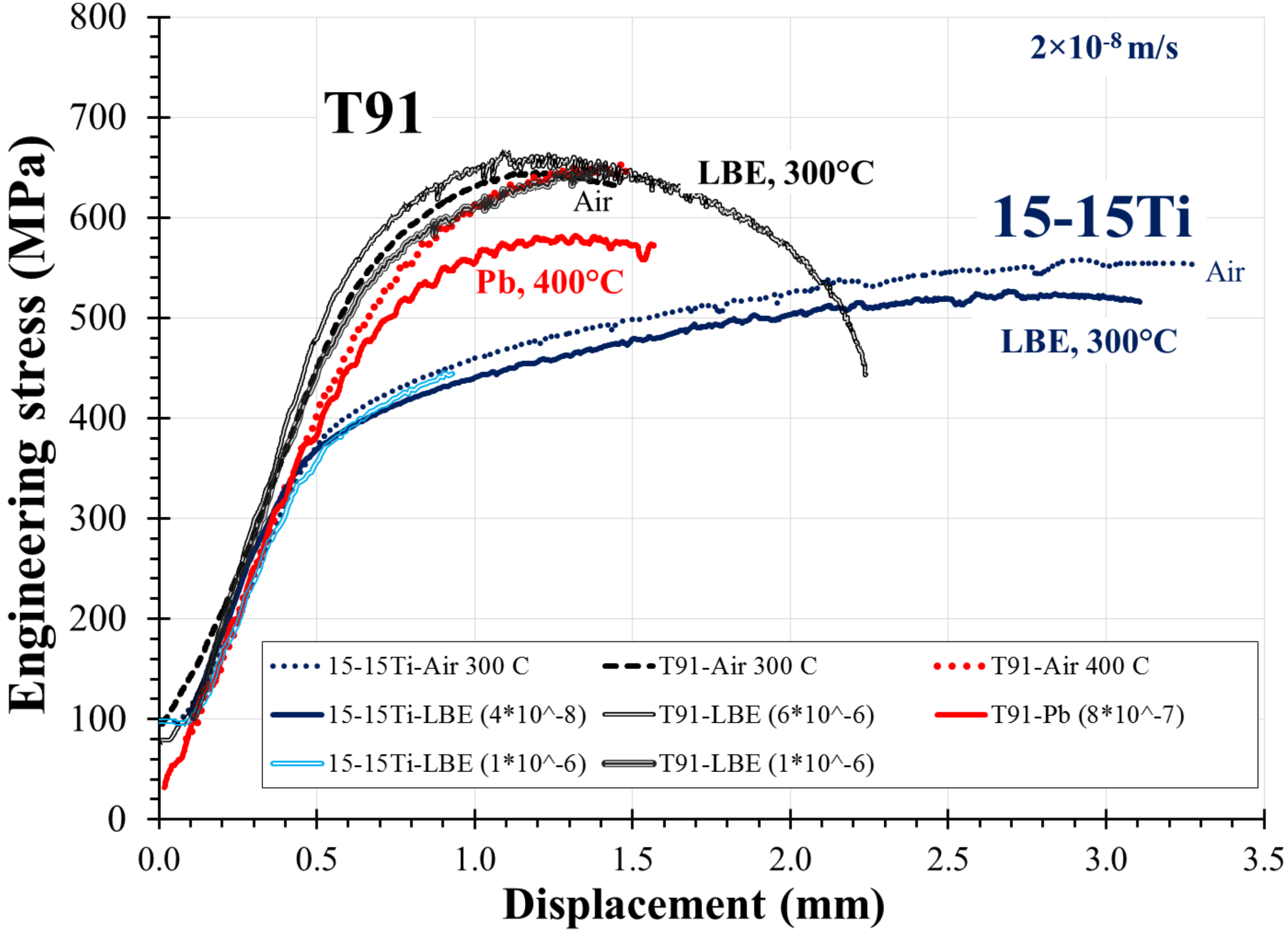
Microscopy observation: SEM + EDS, FIB, TEM

Chemically cleaning in a solution of H_2O_2, CH_3COOH and CH_3CH_2OH (1:1:1)

CERTs

Comparison of stress levels of the three systems

- T91&LBE
- T91&Pb
- 15-15Ti&LBE



T91 & liquid LBE

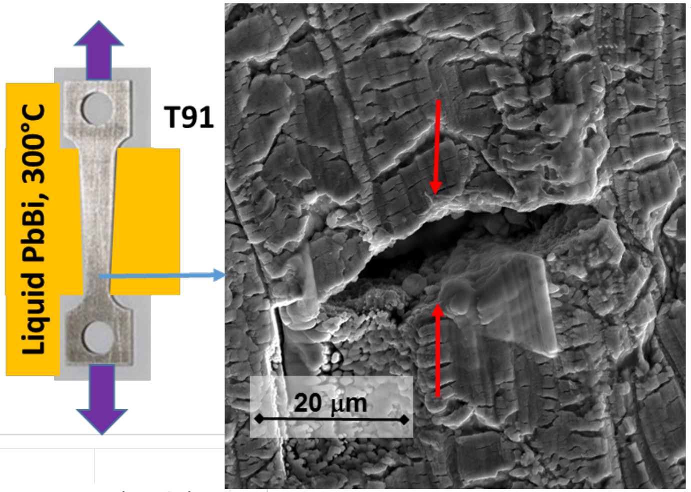
After CERTs,

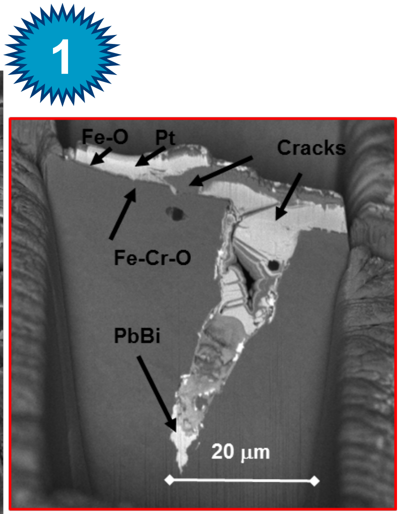
- LME/EAC crack initiation observed in 3 of 5 specimens loaded by R2 - each one had a single crack on the flat surface at minimum cross section

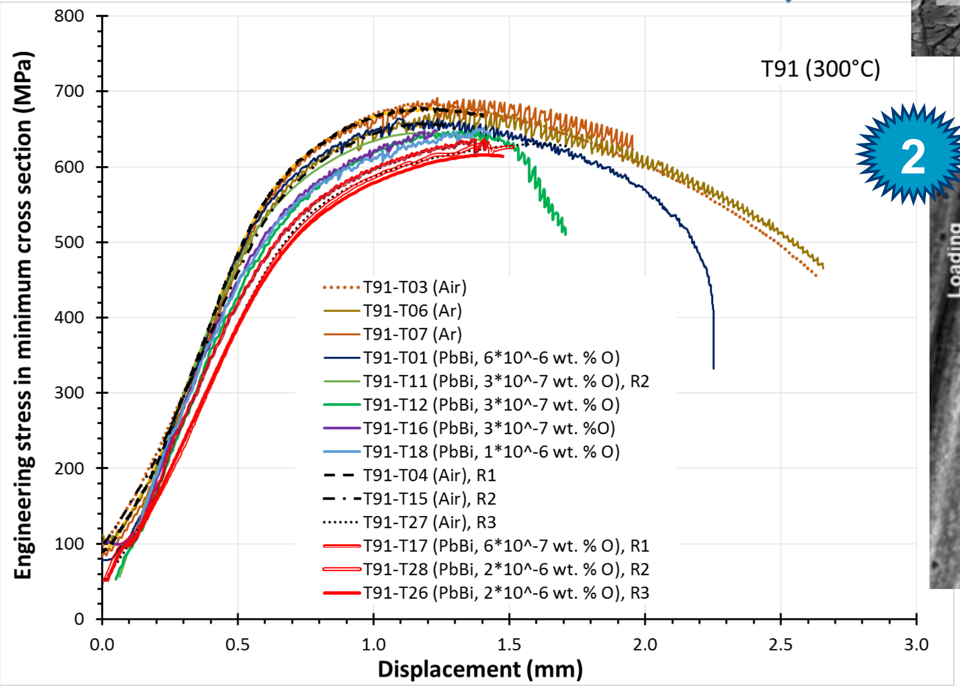
After TPBs in static HLM

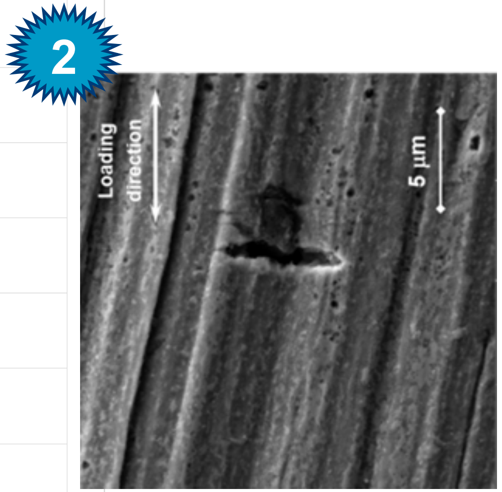
- No cracks

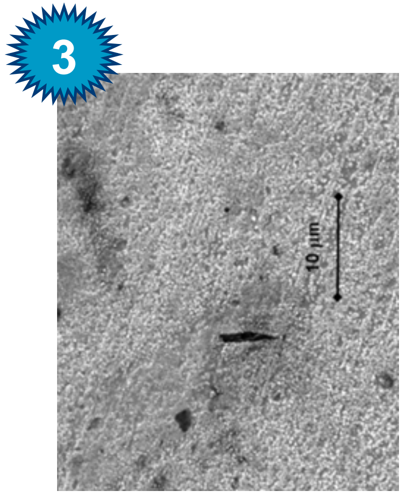
Tapered specimen











Hojna, A.; Hadraba, H; Di Gabriele, F.; Husak, R. Corros. Sci. 131 (2018) 264–277.
Hojna, A.; Di Gabriele, F.; Chocholousek, M.; Halodova, P.; Lorincik, J J. Nucl. Mater. 511 (2018) 459-472.
Halodová, P.; Lorinčík, J.; Hojná, A. Materials 12 (2019) 38.

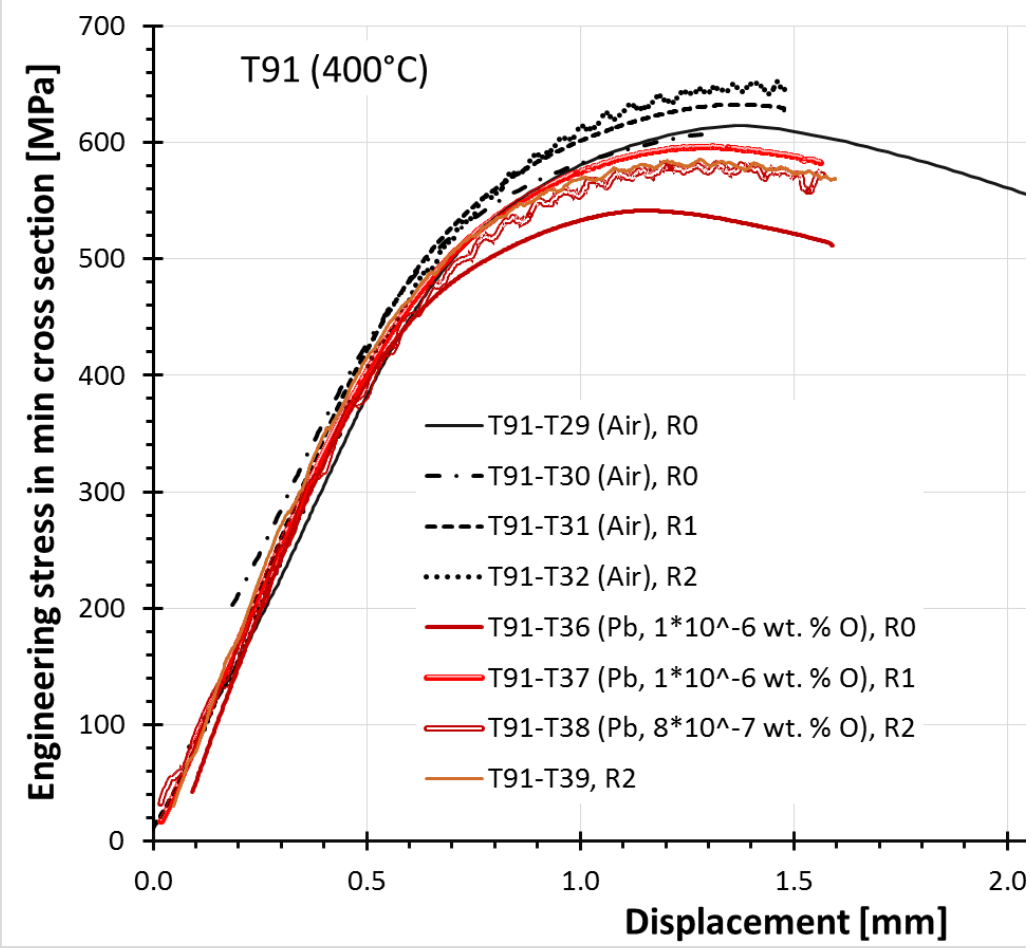
T91 & liquid Pb

After TPBs in static HLM

- No cracks

After CERTs,

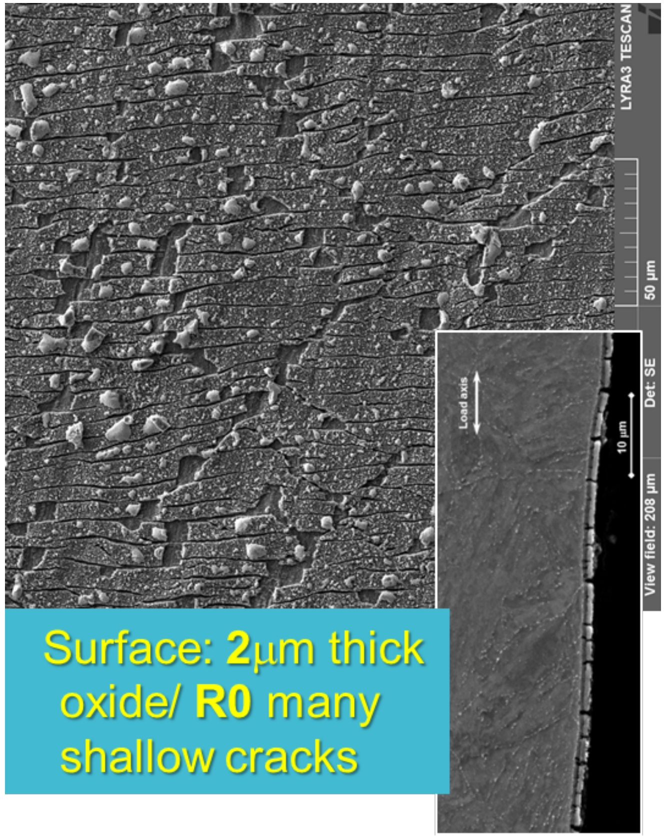
- No LME/EAC crack initiation observed
- 2 specimens loaded by R2



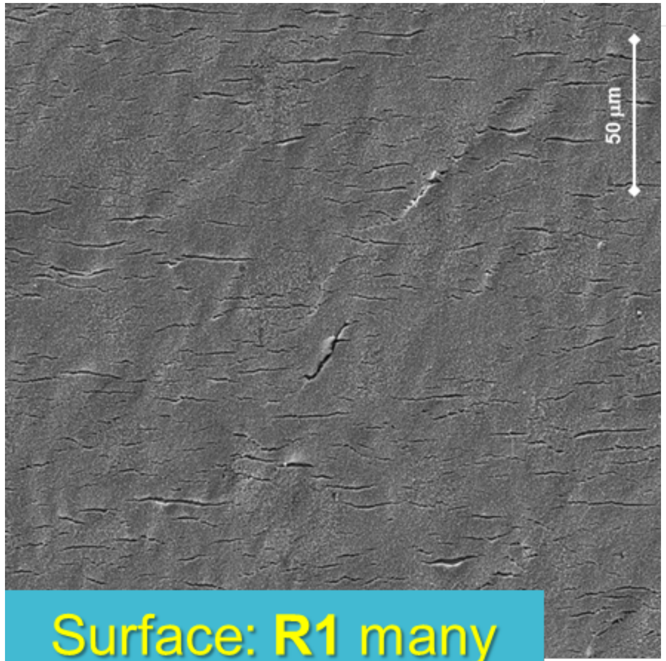
Hojná, A.; Di Gabriele, F.; Chocholoušek, M.; Rozumová, L.; Vít, J. Materials 11 (2018) 1-17

T91 & liquid Pb

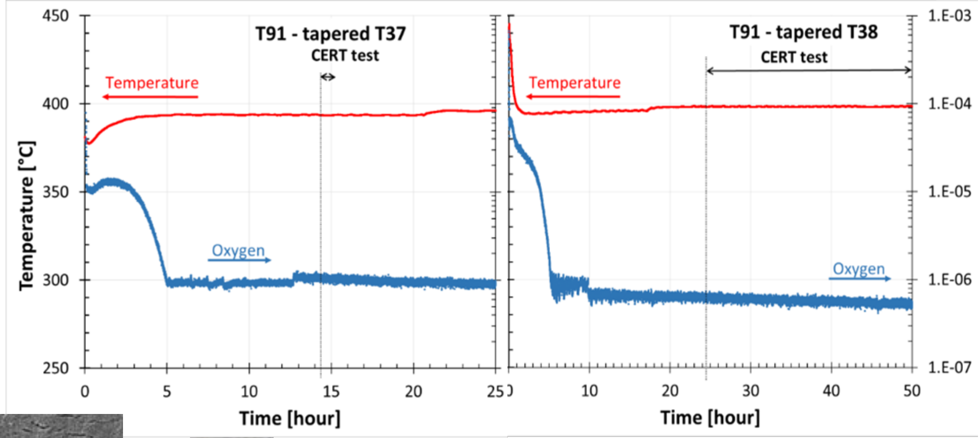
CERT: R0 > R1 > R2

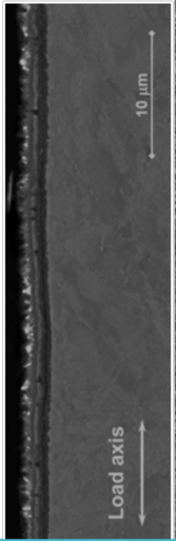


Surface: **2**µm thick oxide/ **R0** many shallow cracks



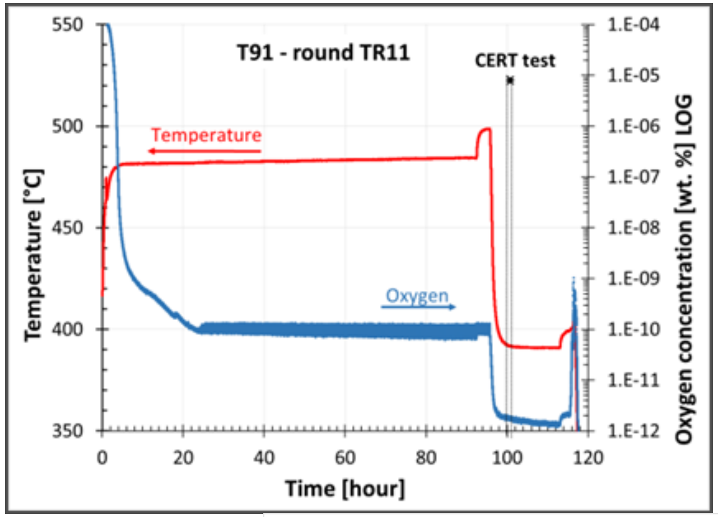
Surface: **R1** many shallow cracks

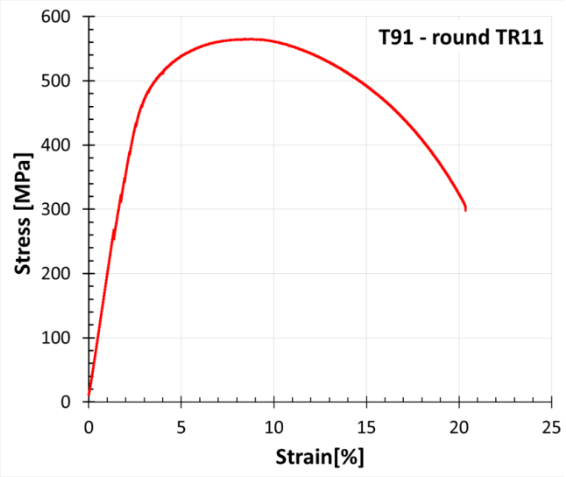




Surface: **2**µm thick oxide/ R2/ rare shallow cracks

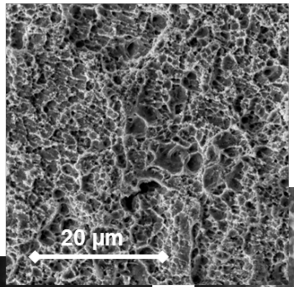
T91 & liquid Pb

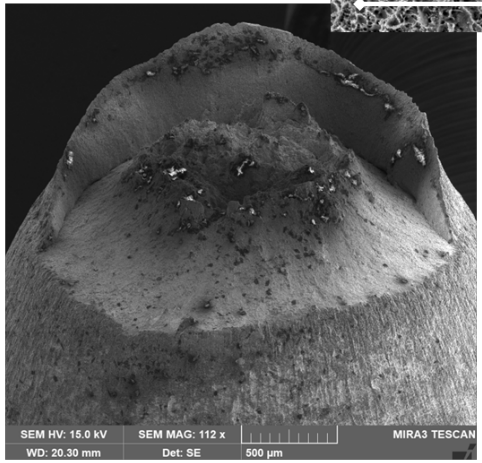


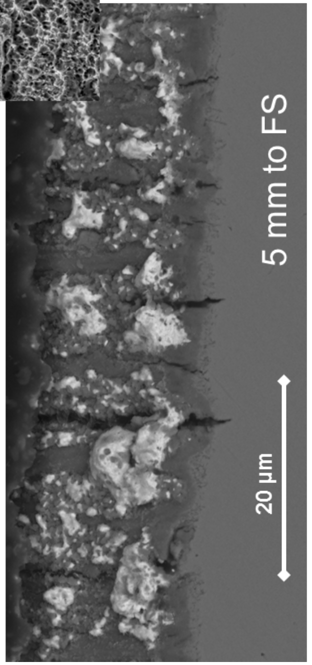


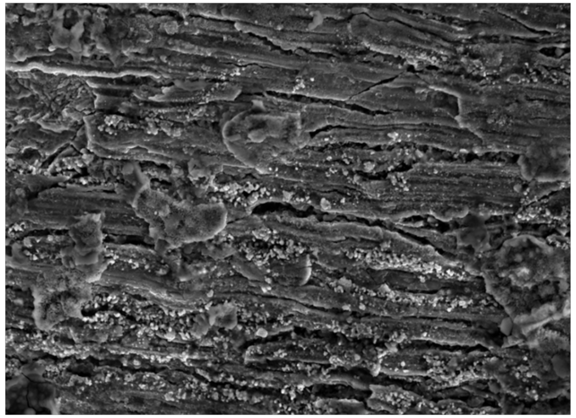
RR test: TR11 (0.12mm/min = 10^{-4}/s)

- **Fracture surface: fully ductile dimples and shear, no LME cleavage-like features**







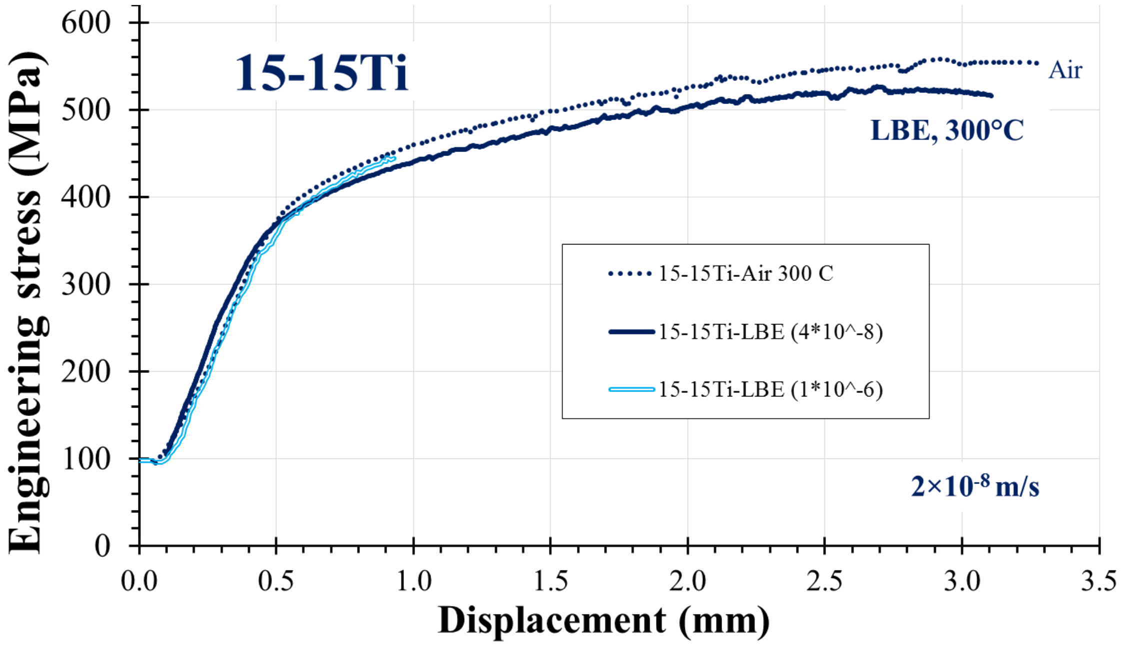


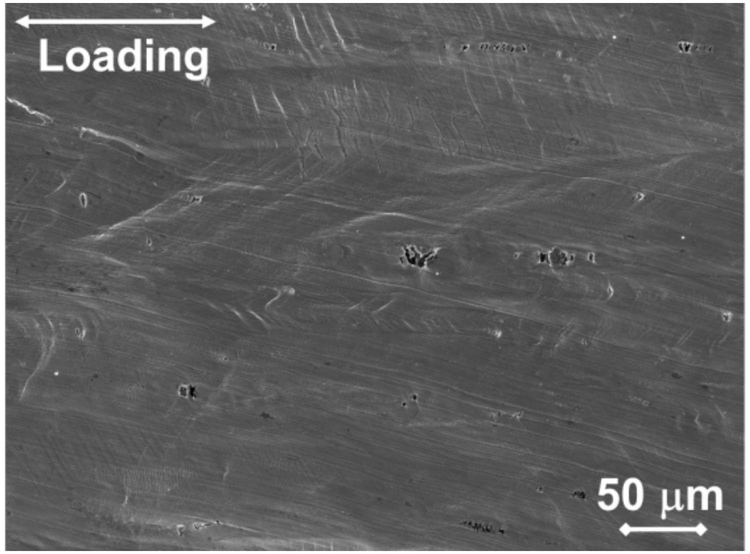
- **Surface: ~5µm thick oxide - many cracks penetrating through**

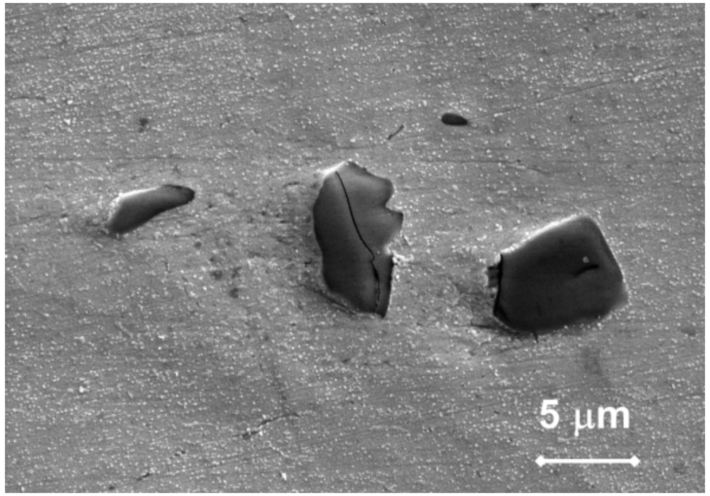
15-15Ti in liquid LBE

After CERTs,

- No LME/EAC crack in 3 specimens loaded by R2





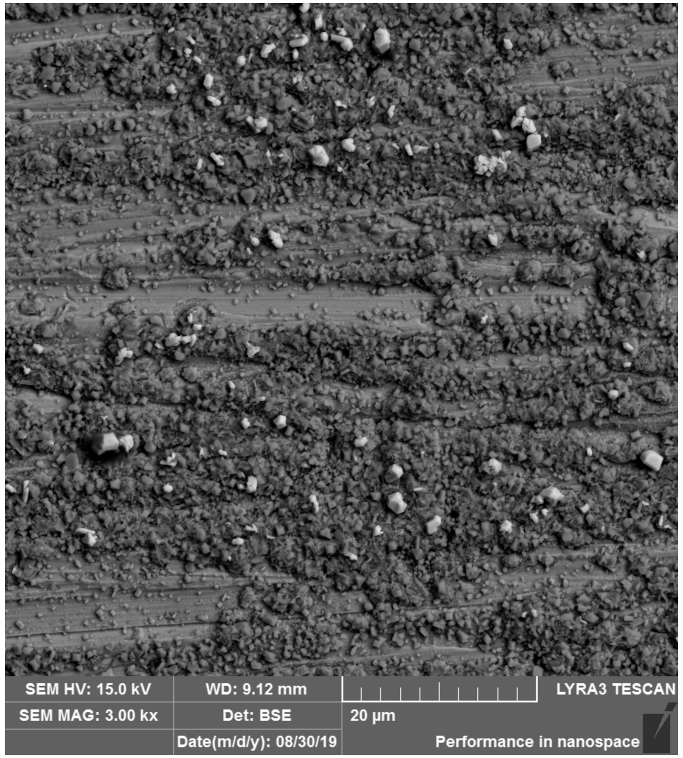


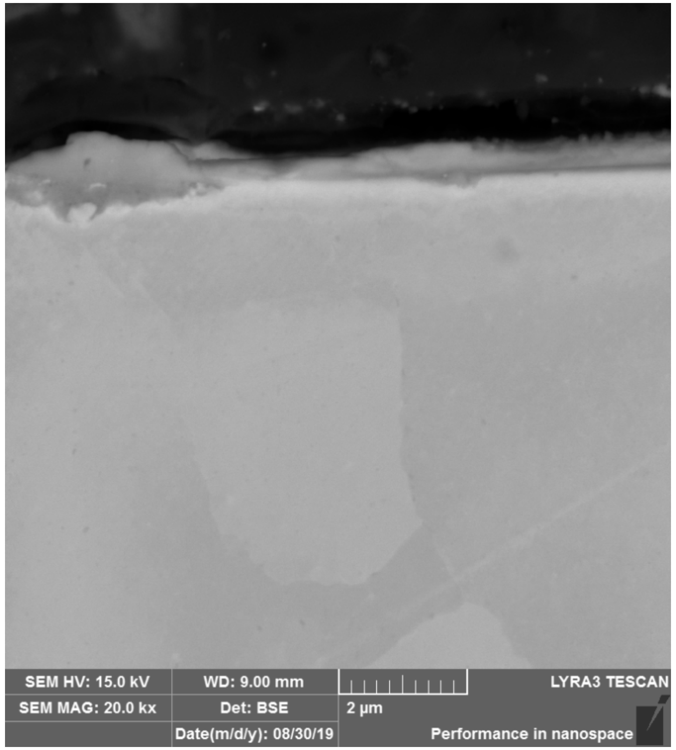
Hojná, A., Di Gabriele, F., Chocholoušek, M., Špirit, Z., Rozumová, L., Journal of Nuclear Engineering and Radiation Science 5 (2019) 1-8.

15-15Ti in liquid Pb

After TPBs in static HLM

- No cracks





Initiation of LME/EAC of T91 in HLM - summary

Stress of LME/EAC initiation is:

Smooth:

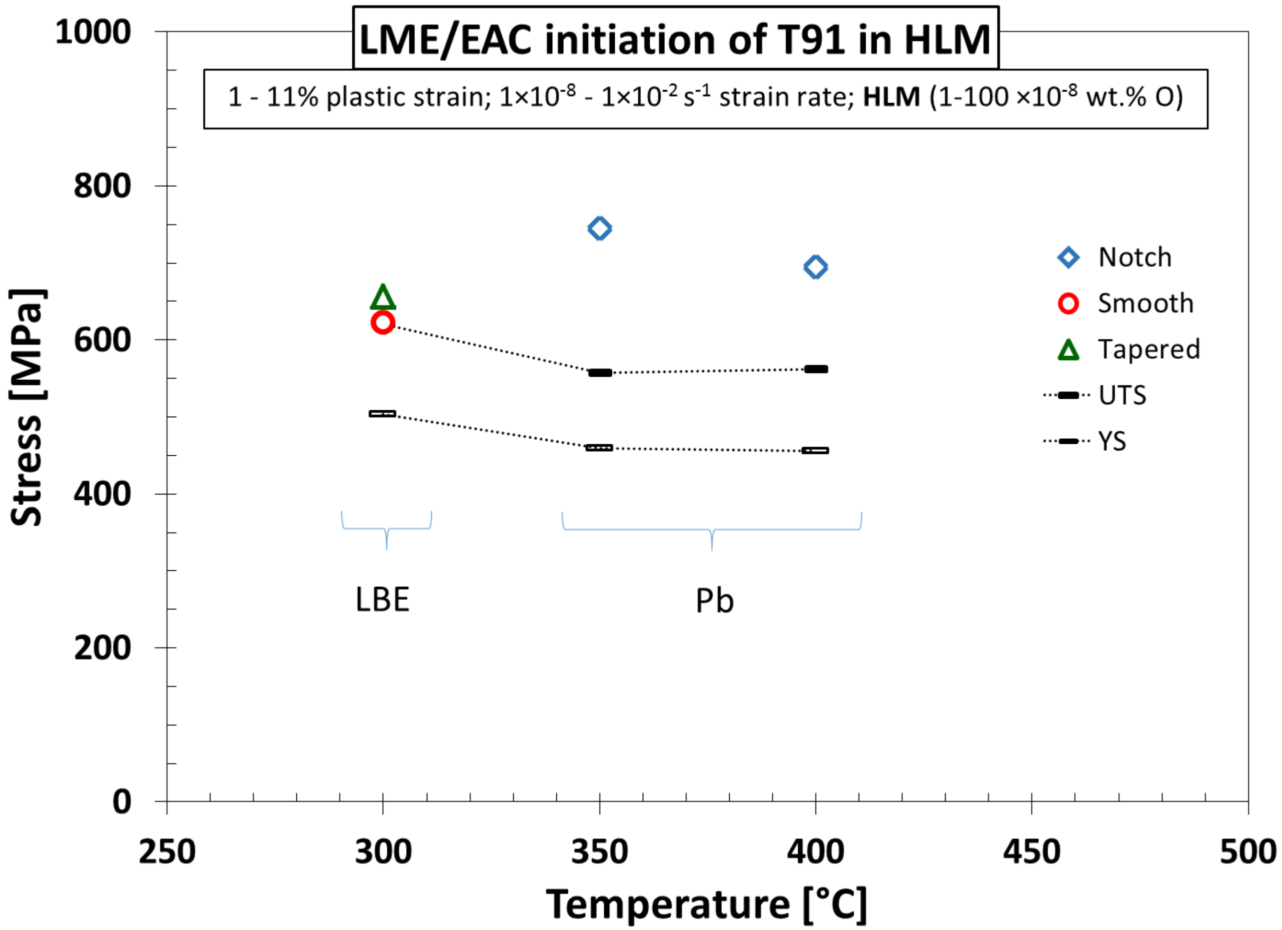
- Nominal stress

Notched:

- Nominal stress × Stress concentration

Tapered:

- Nominal stress at crack site



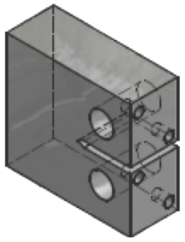
2nd Level testing - Conditions for LME/EAC cracking in HLM: Kinetics

Experimental

Specimens

RCT (thickness 6, 8; Ø15.5 mm)

CT (thickness 12.5 × width 25 mm)



Test technique

Fracture resistance (J integral)

- pre-cracked in air and HLM

Environment

HLM:

Liquid Lead-Bismuth Eutectic (LBE)

T: 350 ºC

O ≈ 4-100 $\times 10^{-8}$ wt. %

Post-test evaluation

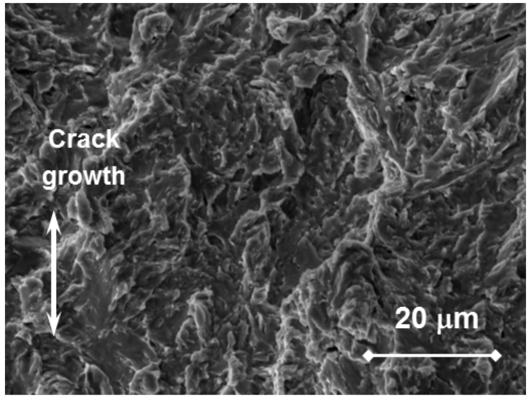
Microscopy observation: SEM + EDS

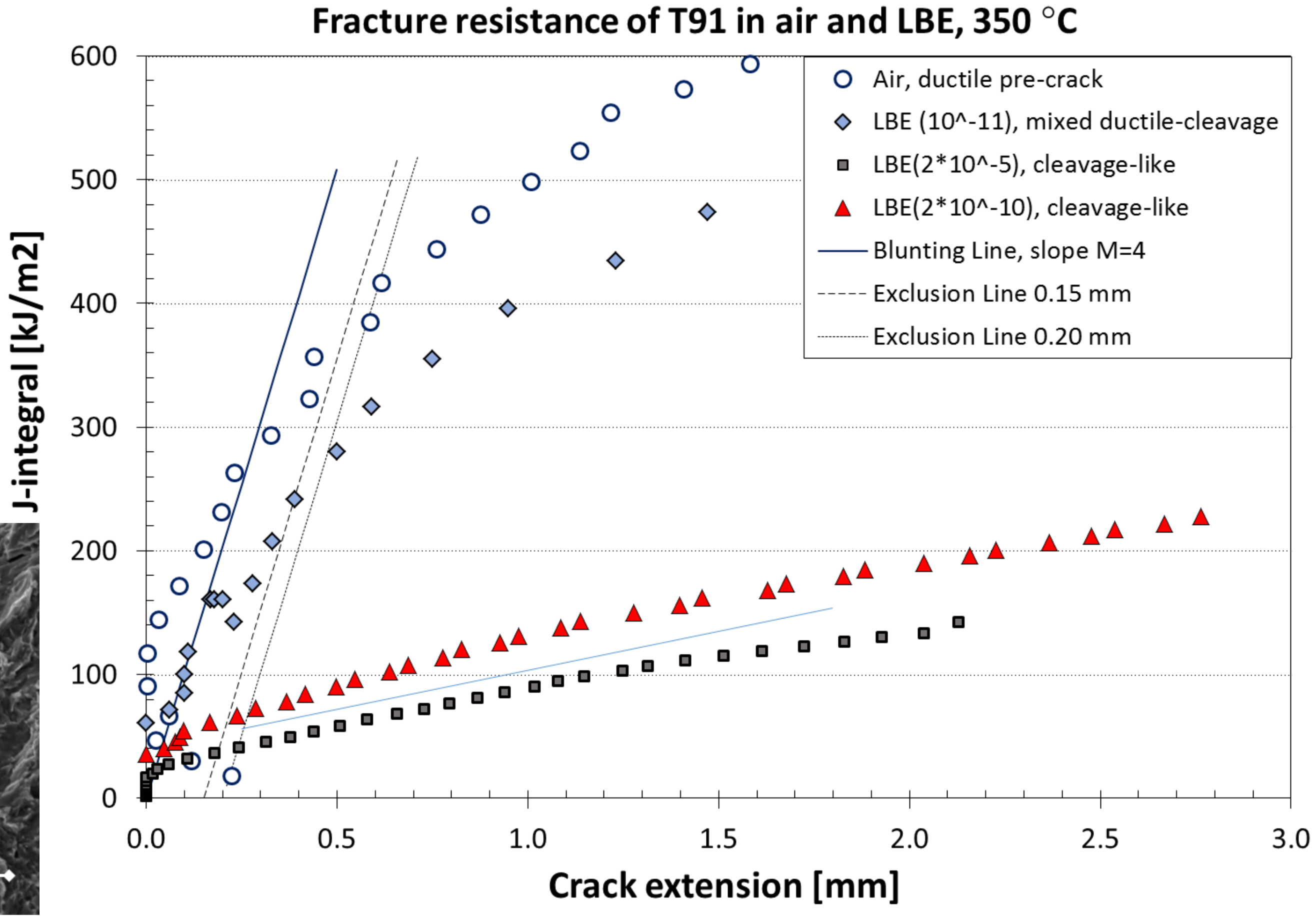
Chemically cleaning in a solution of H_2O_2, CH_3COOH and CH_3CH_2OH (1:1:1)

Fracture resistance of T91 in liquid LBE

Fracture resistance tests (MATTER) using CT with various pre-cracks

- **Mixed ductile-cleavage like**
  - Fatigue in air → pre-cracking in LBE
- **Cleavage-like**
  - Exposed to low O LBE at 450 ºC → pre-cracking in LBE





Hojna, A.; Di Gabriele, F.; Klecka, J. J. Nucl. Mater. 472 (2016) 163–170.
Magielsen, L. et alDeliverable 3.5: "Guidelines, results and evaluations for fracture toughness tests", MATTER (Materials Testing And Rules), 269706 (2015).

Crack Growth Rate of T91 in LBE

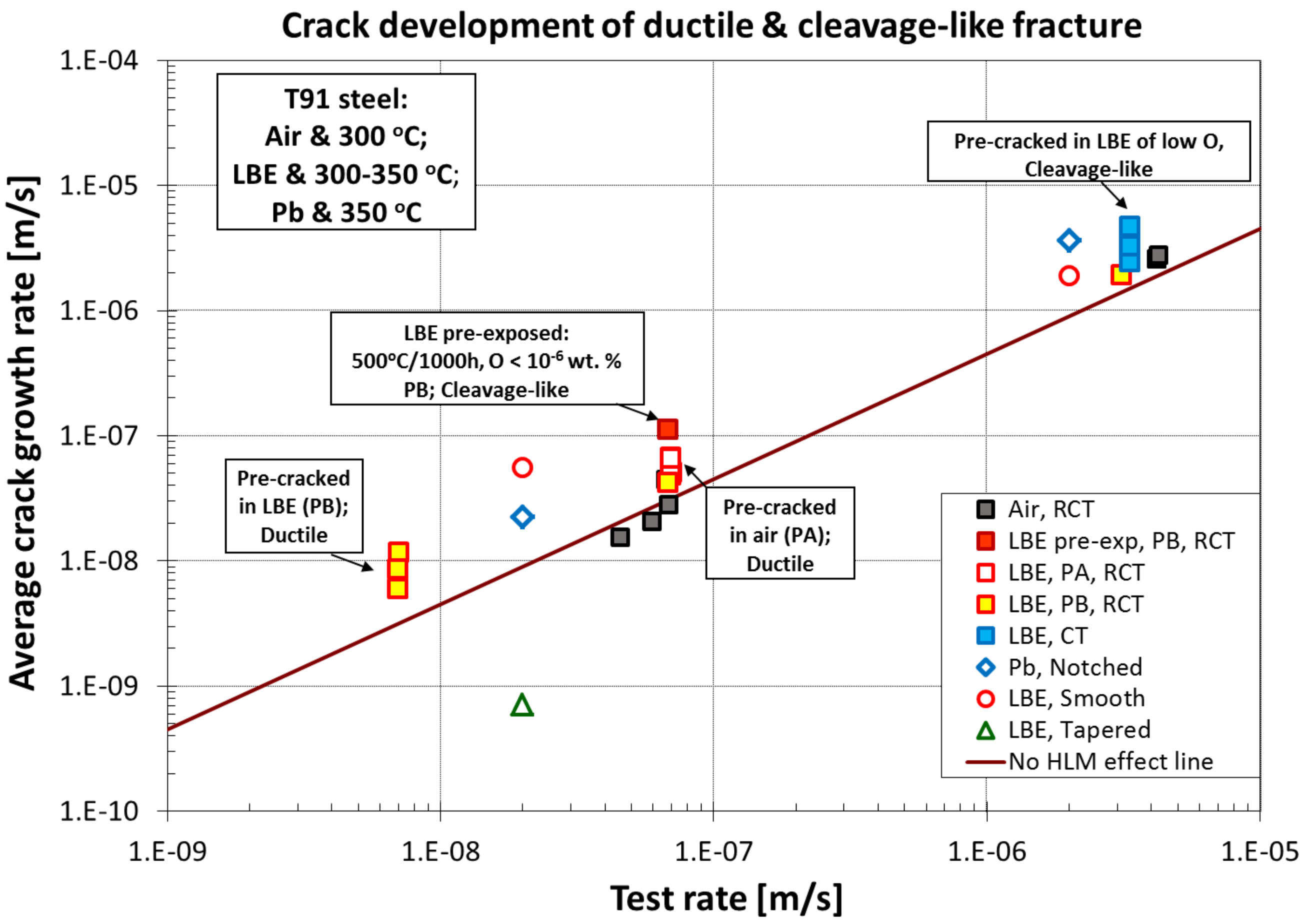
Average crack growth rate measured in experiments with various specimens.

For fully developed crack:

Average crack growth rate
(crack extension/test time)

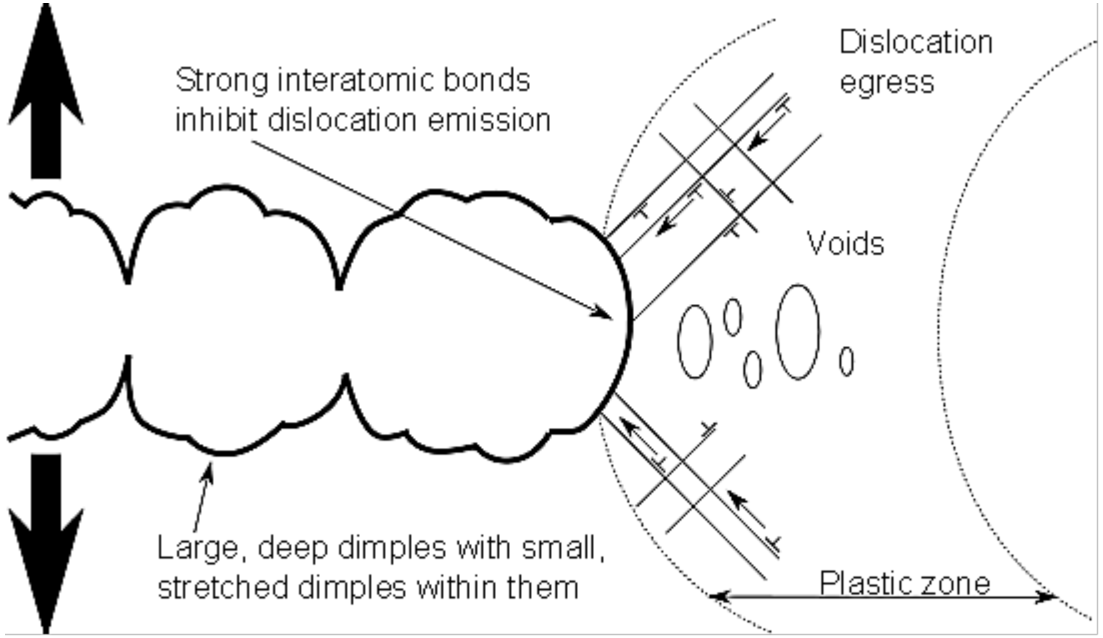
in LBE = max 5× in air

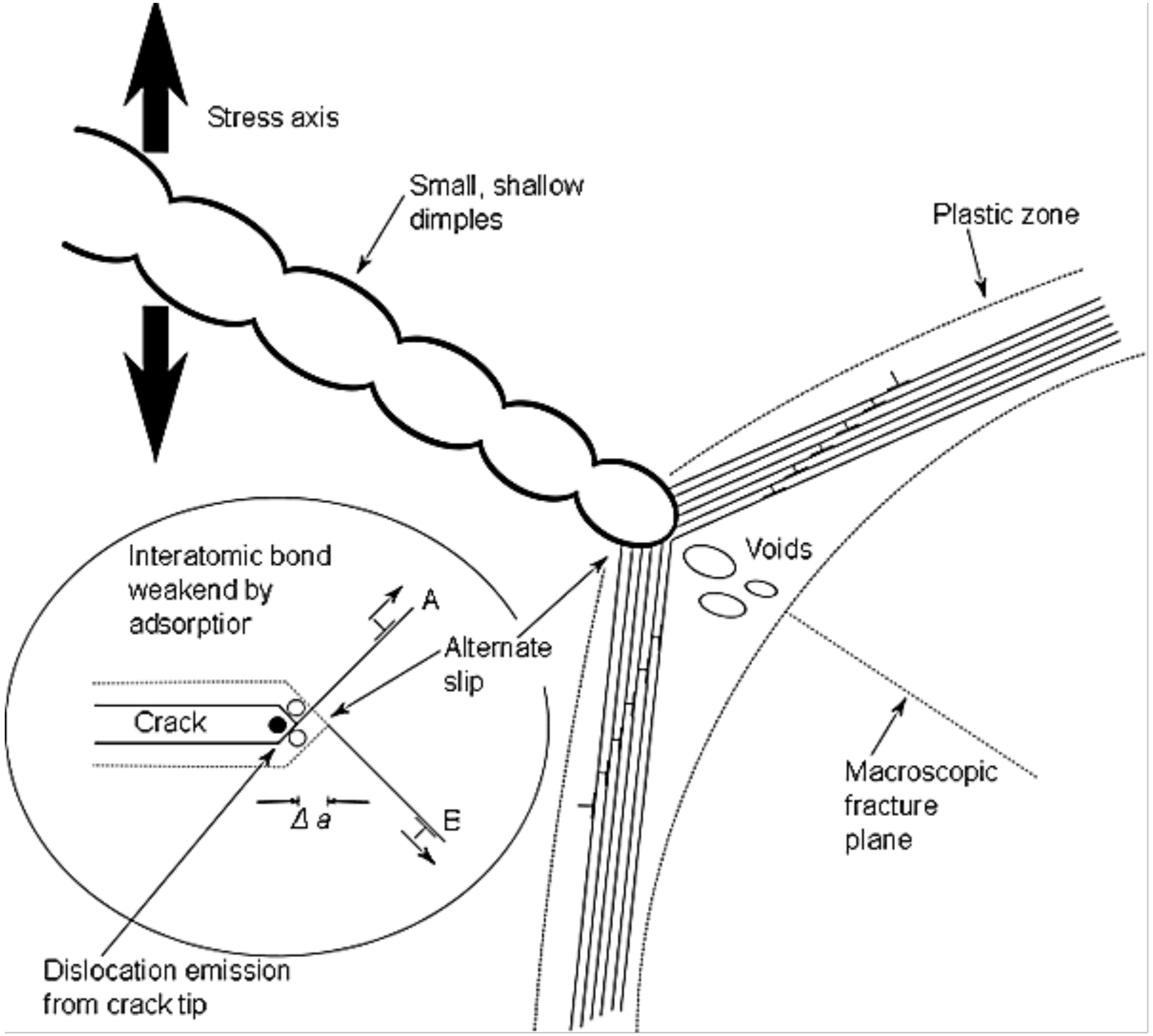
Hojna, A.; Di Gabriele, F., J. Nucl. Mater. 413 (2011) 21–29



LME/EAC mechanism

Ductile fracture





Lynch, S.; SCC: Theory and practice, Woodhead, 2011

Conclusions

Two level assessment of degradation in HLM under stress: **LME ---> LME/EAC**

- Because the HLM atoms do not penetrate into the steels, it is proposed to requalify the degradation mode from LME to EAC.
- Adsorption-induced dislocation-emission mechanism was found to be the best way to describe the observed degradation.

15-15Ti: Initiation testing verified the **immunity to LME/EAC** in LBE & 300°C +Pb & 400°C .

T91 steel showed **sensitivity to LME/EAC**

- LBE &300°C + Pb & 350-400°C: above UTS and high plastic deformation; only with stable failure process.
- Based on the new and past research experience - T91 steel is sensitive to LME/EAC **in low flow HLM**, but only **in beyond design loads** of Gen IV system's components.

Thank you for your attention

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