



# INFLUENCE OF PRE-EXPOSITION IN LIQUID LEAD ON THE MECHANICAL PROPERTIES OF FERRITIC/MARTENSITIC STEEL T91

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April 20-22, 2009

# Influence of Pre-exposition in Liquid Lead on the Mechanical Properties of Ferritic/Martensitic Steel T91



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1. Introduction
2. Experimental
3. Evaluation of experimental
4. Discussion of results
5. Conclusions

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# INTRODUCTION

- The study of mechanical properties and **boundary conditions** necessary to ascertain the sensitivity of T91 to **LME** when specimens loaded in contact with heavy liquid metal is realized in NRI Řež plc.
- **LME – Liquid Metal Embrittlement** ⇒ several different definitions exist; e.g., mechanism of LME means reduction in ductility and fracture toughness of metals when are simultaneously subjected to stresses and wetting by liquid metals [\*].
- **Boundary conditions**
  - metallurgical state,
  - surface state,
  - composition,
  - solubility,
  - temperature,
  - strain rate,
  - stress concentrators etc.

[\*] E. Glickman, “Mechanism of Liquid Metal Embrittlement by Simple Experiments: From Atomistics to Life-time”, In “Multiscale Phenomena in elasticity”, J. Lepinoux, et al. (Eds.), Kluwer Academic Publishers, the Netherlands (2000) pp 383-401.

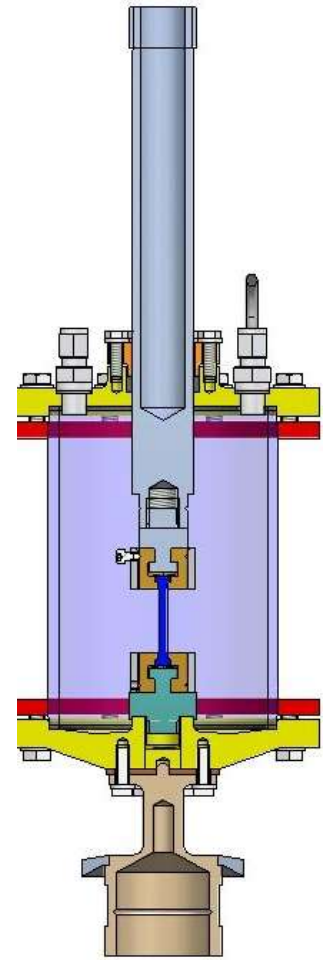


# EXPERIMENTAL

- Experimental facility for mechanical tests of specimens immersed in liquid metals, Pb-Bi and Pb  $\Rightarrow$  experimental cell CALLISTO (see Figure).
- The tensile tests were carried out at 300 °C in air and at 350 °C in liquid lead.
- Specimens from T91 were machined from the plates in the L direction  $\Rightarrow$  **round specimens** with a diameter  $d = 4\text{mm}$  and a gauge length  $l_0 = 20\text{ mm}$ , surface ground up to 600 grit finish.
- Some of the specimens were **pre-exposed in lead at 600 °C for 400 h and  $O_2 = 1 \times 10^{-6}$** .
- Two different strain rates  $10^{-4}$  a  $10^{-6}$  were used.

## Parameters of CALLISTO:

- Temperature operating range cca 200÷700 °C
- Maximum loading capacity = 50 kN
- Specimens and grips are totally immersed into the HLM





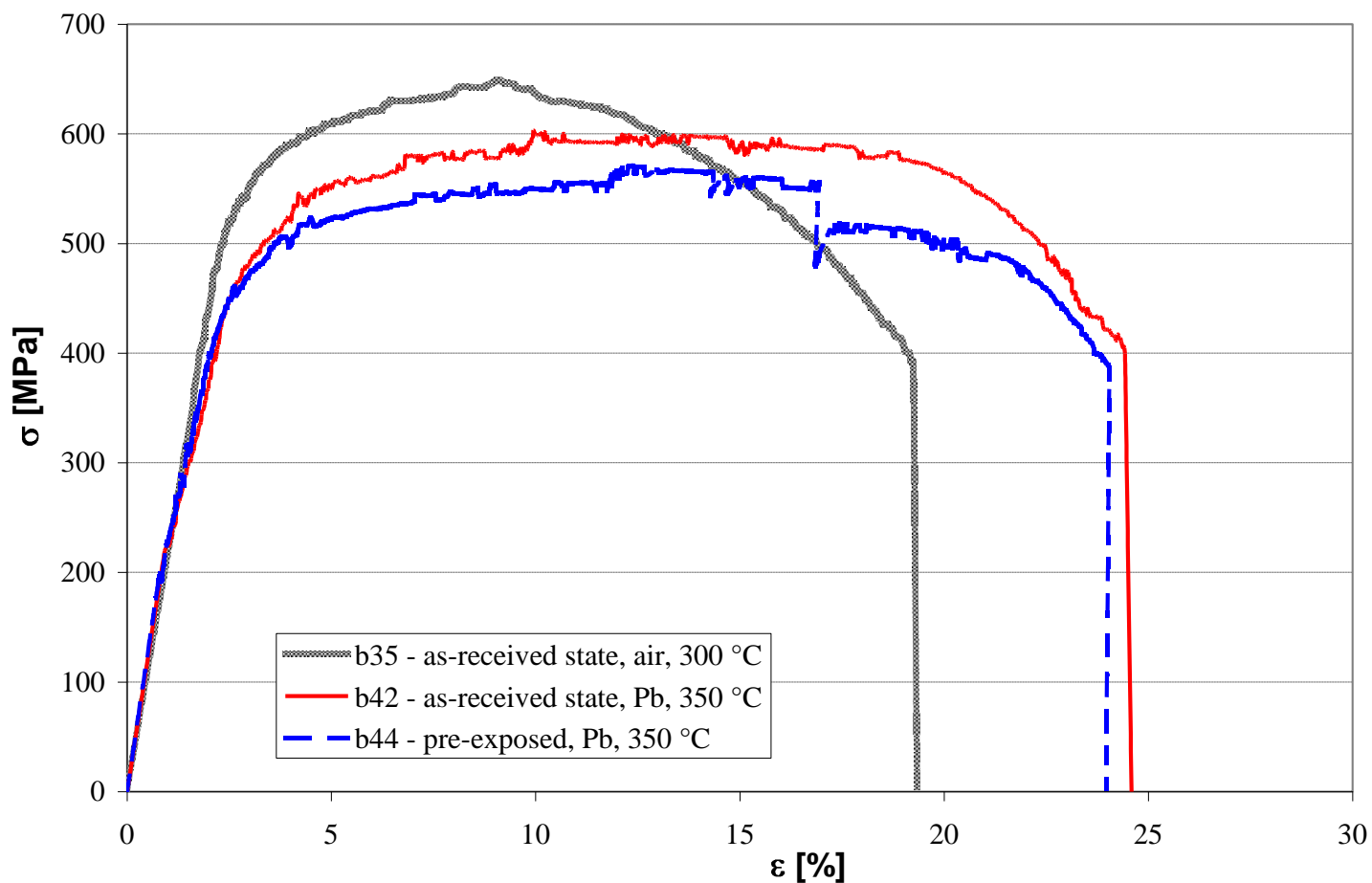
# EXPERIMENTAL

*Summary of the testing conditions for each specimen:*

Spec.	Pre-treat.	Medium	T [°C]	Strain rate [s <sup>-1</sup> ]
B35	as/rec	air	300	1x10 <sup>-6</sup>
B36	as/rec	air	300	1x10 <sup>-4</sup>
B41	as/rec	Pb	350	1x10 <sup>-4</sup>
B42	as/rec	Pb	350	1x10 <sup>-6</sup>
B43	exp. Pb	Pb	350	1x10 <sup>-4</sup>
B44	exp. Pb	Pb	350	1x10 <sup>-6</sup>

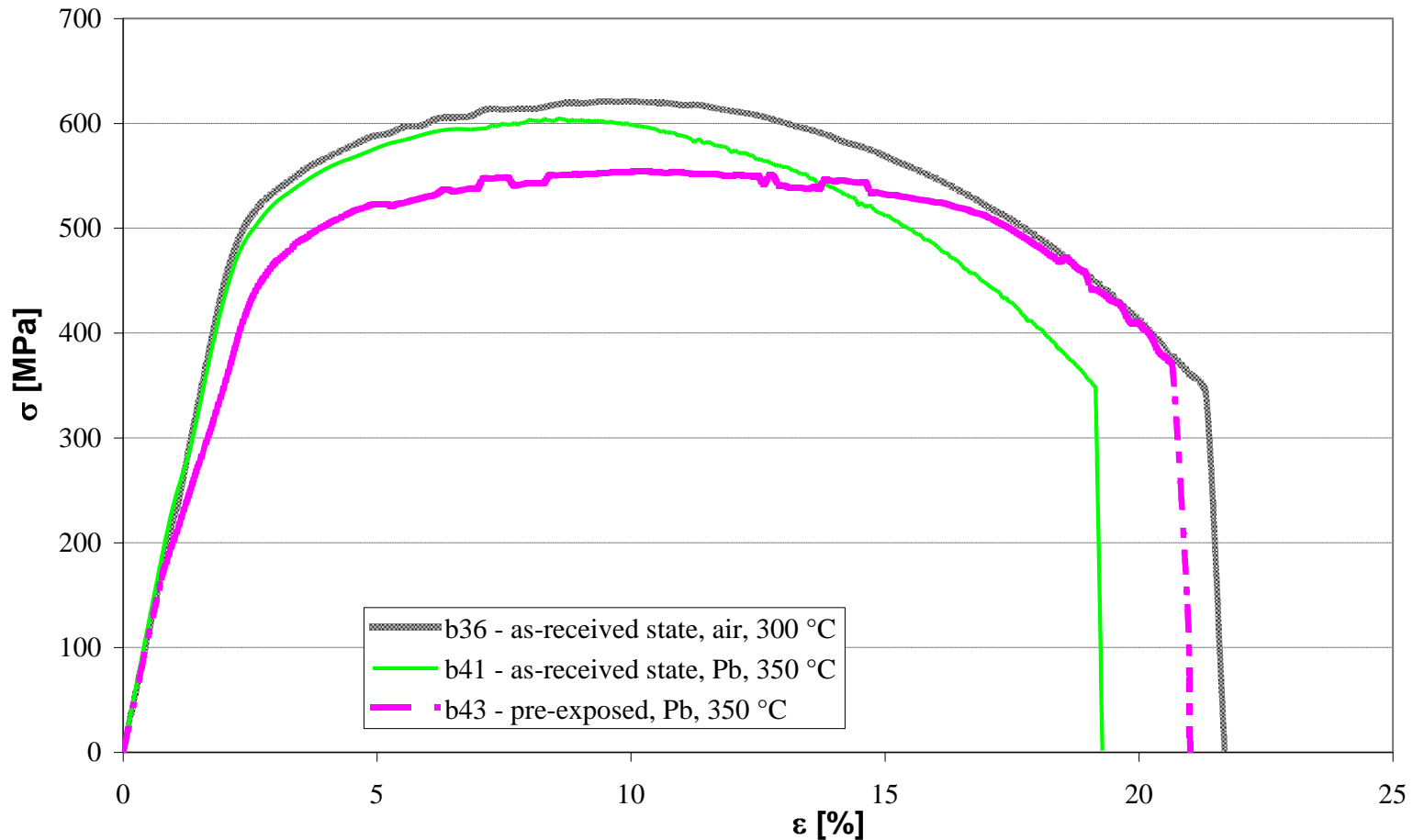


# EXPERIMENTAL - results



*Stress-strain curves for specimens tested at strain rate  $10^{-6} \text{ s}^{-1}$*

# EXPERIMENTAL - results



*Stress-strain curves for specimens tested at strain rate  $10^{-4} s^{-1}$*

## Summary of tensile tests results:

Spec.	Conditions				RESULTS				
	pre-treat	environ	T [°C]	strain rate [s <sup>-1</sup> ]	Z%	σ <sub>y</sub> [MPa]	σ <sub>uts</sub> [MPa]	ε <sub>uts</sub> [%]	ε <sub>tot</sub> [%]
B35	as/rec	air	300	1x10 <sup>-6</sup>	71	575	646	9,3	19,3
B42	as/rec	Pb	350	1x10 <sup>-6</sup>	70	478	603	10,0	24,6
B44	exp. Pb	Pb	350	1x10 <sup>-6</sup>	74	450	571	12,4	24,0
B36	as/rec	air	300	1x10 <sup>-4</sup>	73	499	620	10,2	21,7
B41	as/rec	Pb	350	1x10 <sup>-4</sup>	75	490	604	8,7	19,3
B43	exp. Pb	Pb	350	1x10 <sup>-4</sup>	75	451	555	10,2	21,0

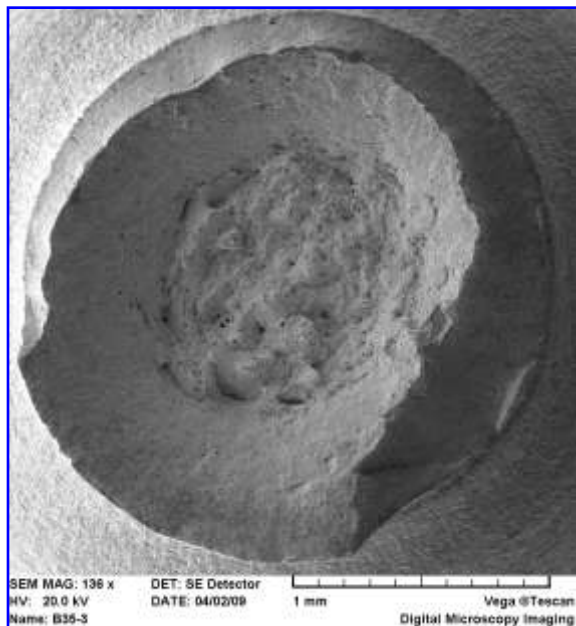
- *The reduction of area did not changed markedly in both environments and for specimens pre-exposed in Pb, as well as.*
- *The slight decrease of engineering values, such as yield stress and UTS compared to the values measured in air and Pb without pre-exposition.*
- *Elongation to rupture is increased for lower strain rates 10<sup>-6</sup> s<sup>-1</sup> compared with the rates 10<sup>-4</sup> s<sup>-1</sup>.*



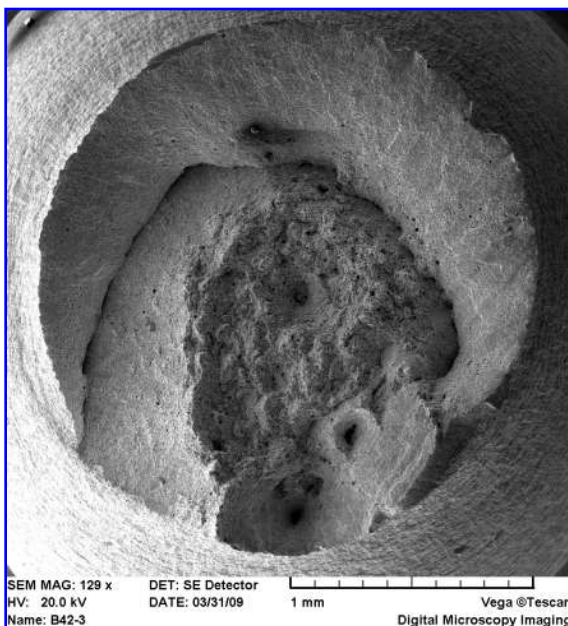
# EVALUATION OF EXPERIMENTAL

*Fractographic analysis*

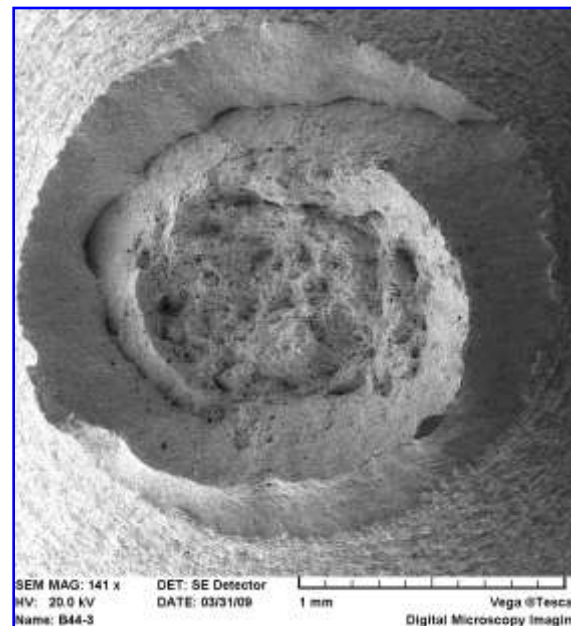
*Specimens tested at strain rate  $10^6 \text{ s}^{-1}$*



As-received state,  
air, 300 °C



As-received state,  
Pb, 350 °C

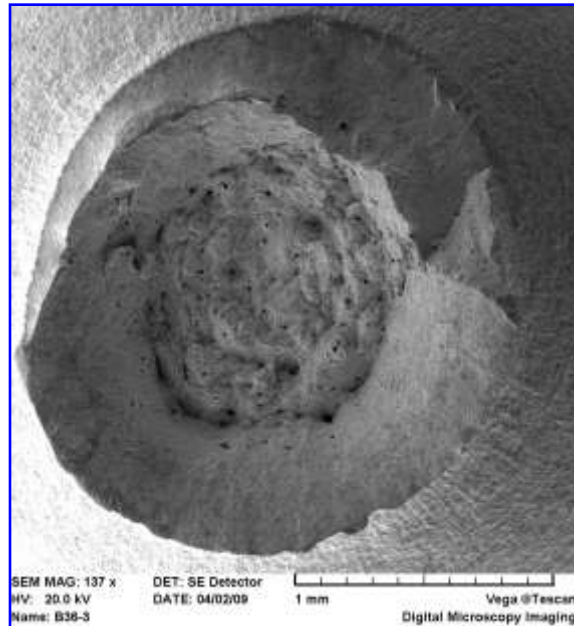


Pre-exposed in Pb  
at 600 °C for 400 h,  
Pb, 350 °C

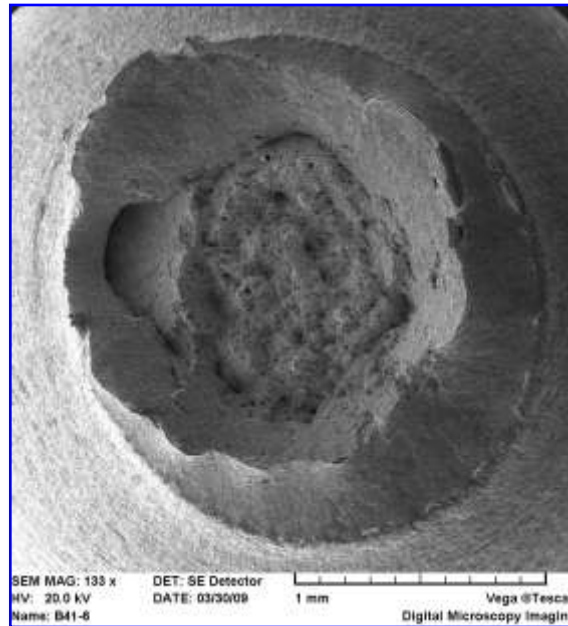
# EVALUATION OF EXPERIMENTAL

## Fractographic analysis

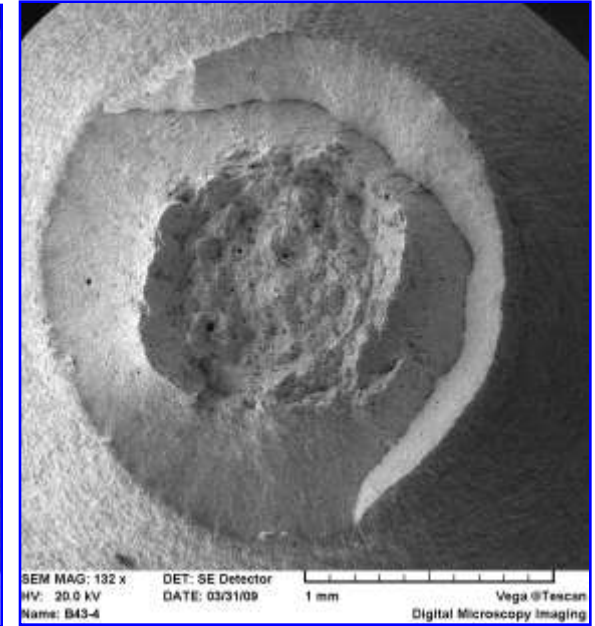
Specimens tested at *strain rate*  $10^4 \text{ s}^{-1}$



As-received state,  
air, 300 °C



As-received state,  
Pb, 350 °C

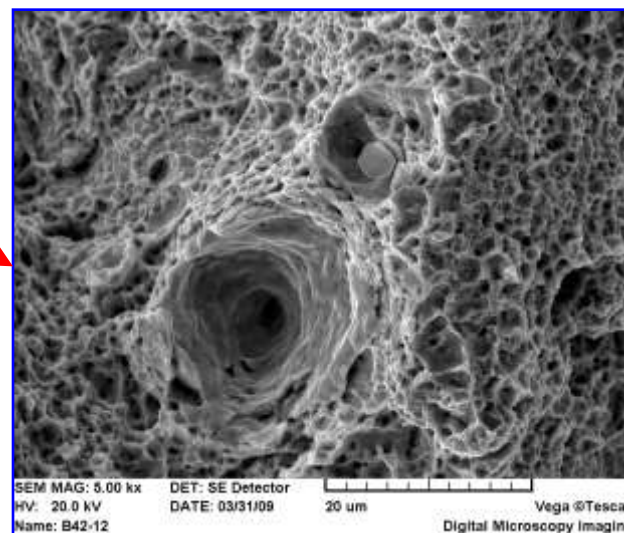
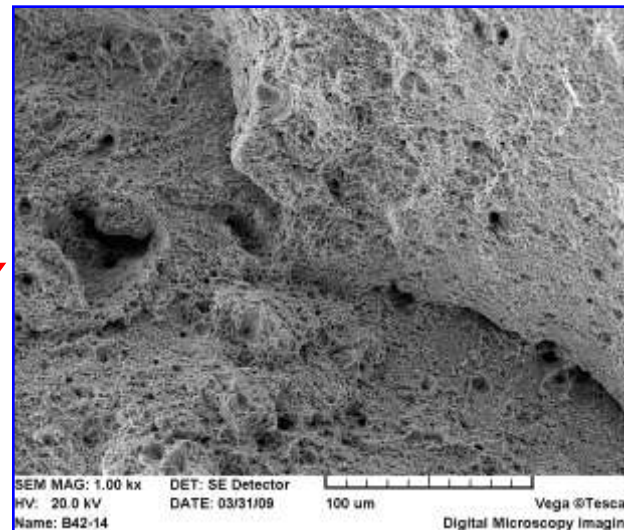
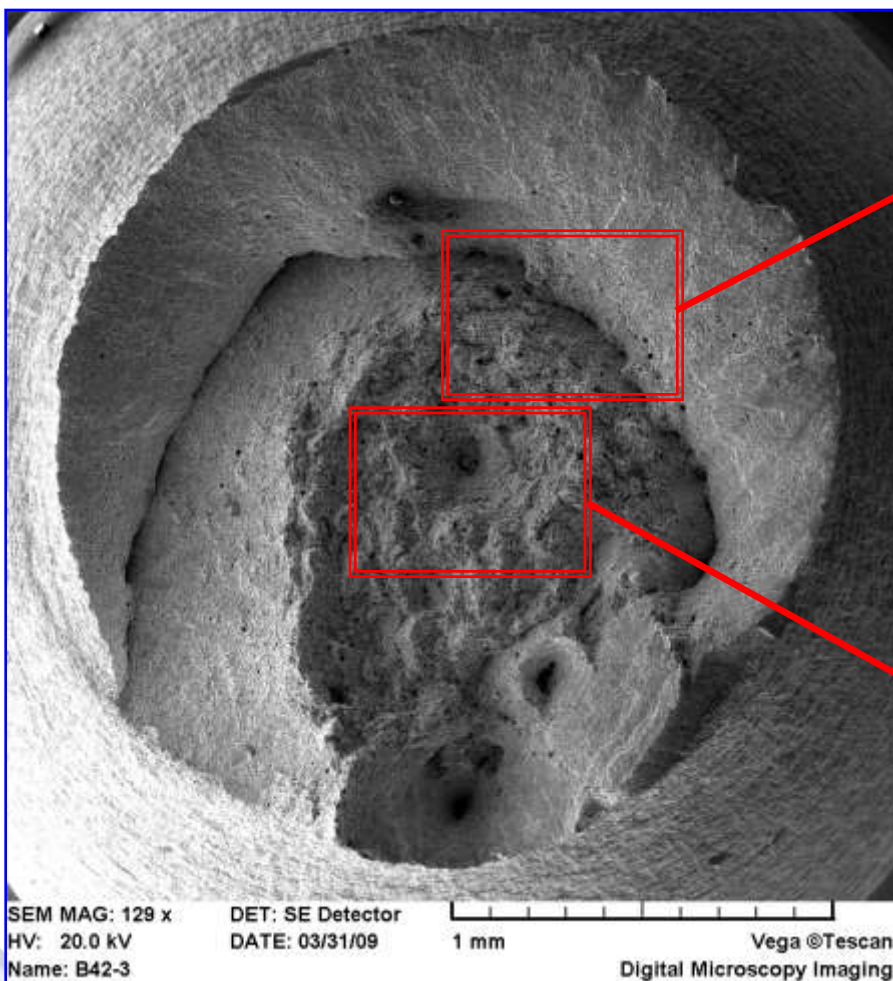


Pre-exposed in Pb at  
600 °C for 400 h,  
Pb, 350 °C



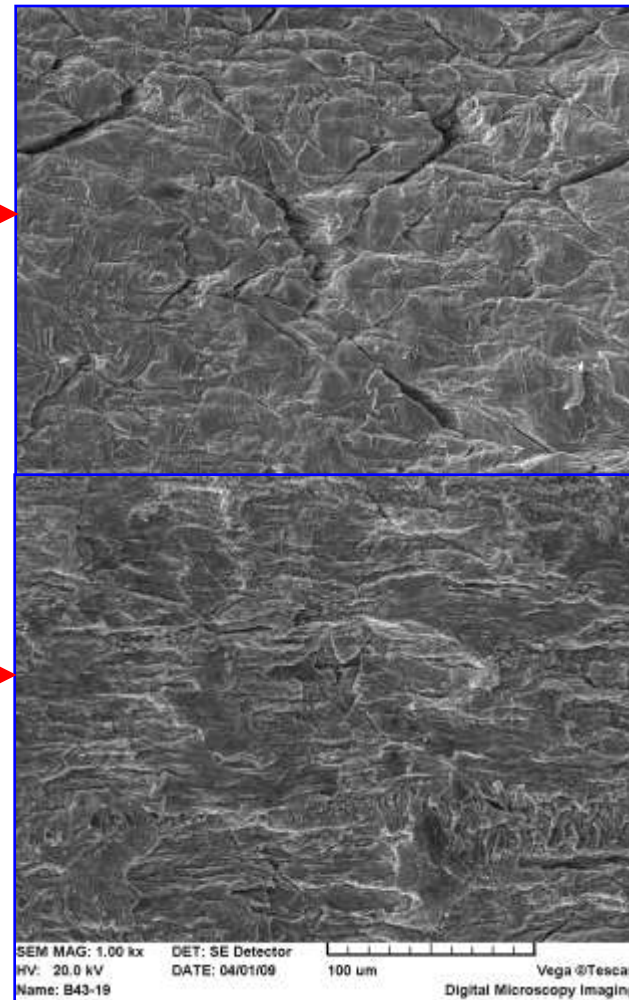
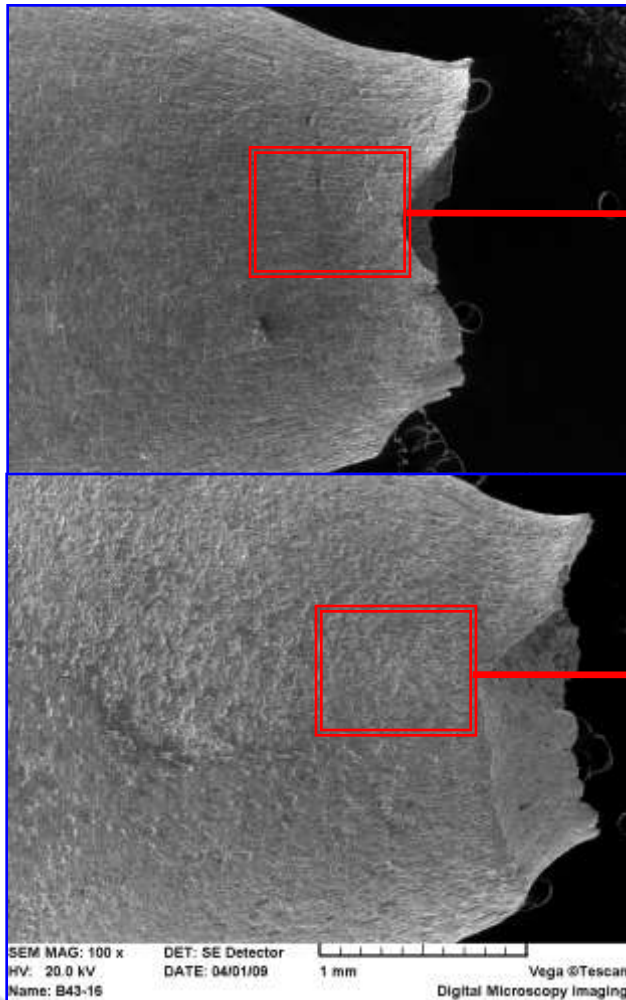
# EVALUATION OF EXPERIMENTAL

## Fractographic analysis



# EVALUATION OF EXPERIMENTAL

*Fractographic analysis of specimens' surfaces – Pb, 350 °C, strain rate  $10^{-4} s^{-1}$*



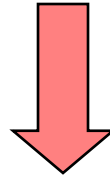
As-received state

Pre-exposed in Pb



## DISCUSSION OF RESULTS

*Aim:* The study of sensitivity of ferritic-martensitic 9 Cr steel to LME when is in contact with liquid lead at 300÷350 °C, different strain rates and with or without pre-exposition in Pb.



By comparison of results for specimens in as-received state tested in Pb and specimens that were pre-exposed in convection loop, it was possible to evaluate the influence of pre-exposition on the mechanical properties changes.

All results from the tensile tests were complemented with the results of fractographic analysis in order to have a direct correlation between the mechanical properties measured and the characteristic fracture features.

## DISCUSSION OF RESULTS

- ❑ The oxide layers on the surface of all tested specimens were found out → the oxygen dissolved in liquid Pb was sufficient to develop a protective oxide layer on the steel that prevented direct contact between the steel and heavy liquid metal.
- ❑ **At lower strain rates  $10^{-6} \text{ s}^{-1}$** , the increase in ductility of specimens tested in Pb (in as-received state and with pre-exposition) and the decrease of ultimate tensile strength,  $\sigma_{\text{UTS}}$ , was observed. By comparison of results obtained in air and in Pb with previous pre-exposition in Pb, the decrease in  $\sigma_{\text{UTS}}$  was 11.5%.
- ❑ Different results were obtained for **higher strain rates  $10^{-4} \text{ s}^{-1}$**  → lower elongation to rupture was found out for specimen tested in Pb in as-received state. The decrease in  $\sigma_{\text{UTS}}$  of pre-exposed specimen tested in Pb in comparison of specimen tested in air was also almost 11%.
- ❑ The reduction of area,  $Z$ , was for all specimens in a range of 70÷75%.



## DISCUSSION OF RESULTS

- ❑ The results of fractographic analysis correlate with the obtained stress-strain curves.
- ❑ All specimens failed in ductile mode, evidencing that the material was beyond the UTS point when it normally started necking and cracking.
- ❑ However, the difference between the surfaces of specimens with and without pre-exposition was observed by SEM → in longer distance from fracture, the surface affected by pits with character of selective corrosion was observed → this kind of surface damage was possibly caused due to the previous specimens pre-exposition in Pb → further investigation will be connected.



# CONCLUSIONS

- It was proved that the pre-exposition in Pb influences the engineering values, i.e., the decrease of yield stress and ultimate tensile strength in comparison with the values obtained in air and Pb (specimens without pre-exposition).
- The increase in ductility, i.e., the increase in elongation to rupture, was found out for specimens immersed in Pb at 350 °C and strain rate  $10^{-6} \text{ s}^{-1}$  compared with the same test in air.
- Generally, the elongation to rupture of specimens immersed in Pb is also influenced by the changes in strain rate. For lower strain rates  $10^{-6} \text{ s}^{-1}$ , elongation to rupture is increased than for higher rates  $10^{-4} \text{ s}^{-1}$ .



# Thank you for your attention



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