

Influence of oxide layer strength on LME susceptibility of Si enriched steels

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¹ LANL, USA

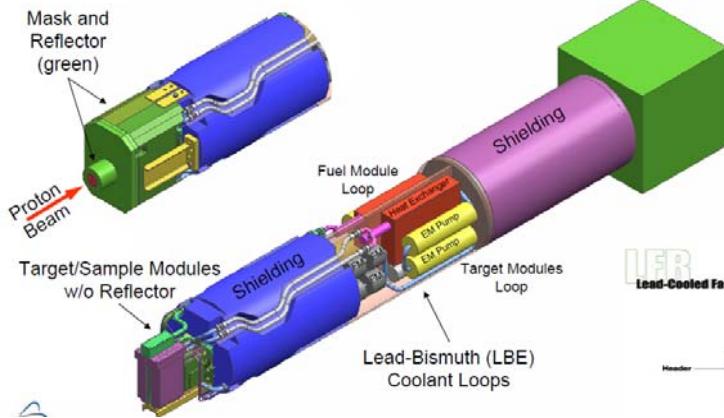
² SCK•CEN, Belgium

Outline

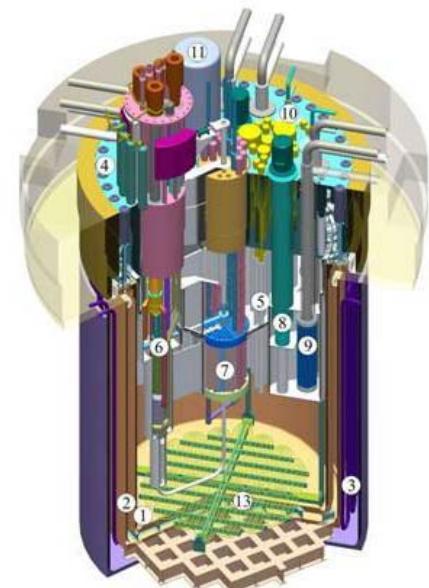
- **Introduction**
- **Materials**
- **SSRT in LBE and Fracture Surface Analysis**
- **Nano-indentation of oxide layers formed in LBE**
- **Conclusions & Outlook**

Introduction

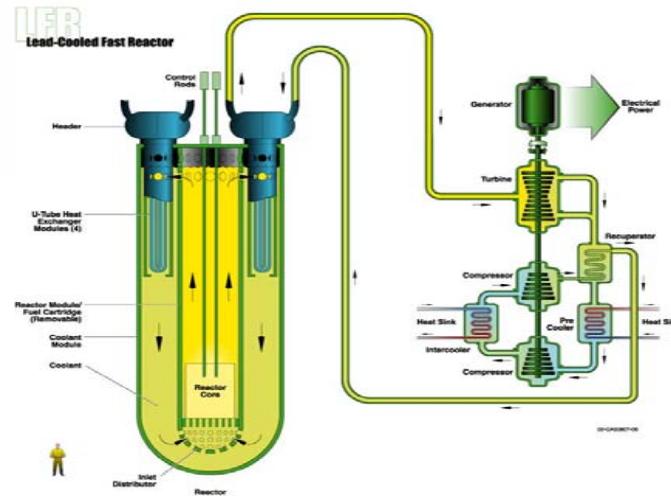
MTS



MYRRHA (ADS)

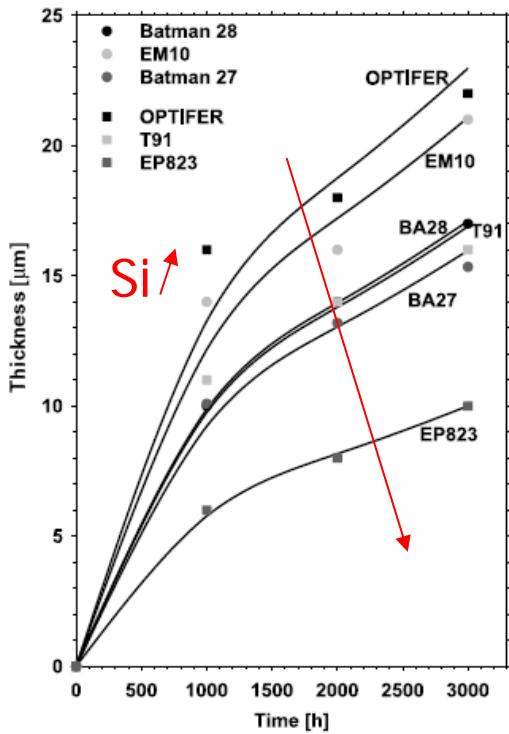


LFR



corrosion & mechanical integrity!

Ferritic-martensitic steels



	Cr	Ni	Mo	Mn	V	Nb	W	Ti	Si	C
<i>Austenitic steel</i>										
1.4970	15.4	14	1.8	2.4	—	—	—	0.4	0.5	0.12
AISI 316L	17.3	12.1	2.31	1.8	—	—	—	—	0.35	0.02
<i>Martensitic steel</i>										
Optifer IVc	9.1	—	—	0.52	0.22	—	1.4	—	—	0.12
EM10	9.0	0.1	1.0	0.5	—	—	—	—	0.3	0.1
T91	8.26	0.13	0.95	0.38	0.20	0.075	—	—	0.43	0.105
Batman 28	8.94	0.05	0.01	3.51	0.24	0.01	1.51	0.01	0.32	0.09
Batman 27	9.0	0.07	0.01	3.1	0.21	0.01	1.45	0.20	0.49	0.1
EP823	12	0.89	0.7	0.67	0.43	0.4	1.2	—	1.8	0.14

F. Barbier, G. Benamati, C. Fazio, A. Rusanov, J. Nucl Mater, 295 (2001) 149

Ferritic steels

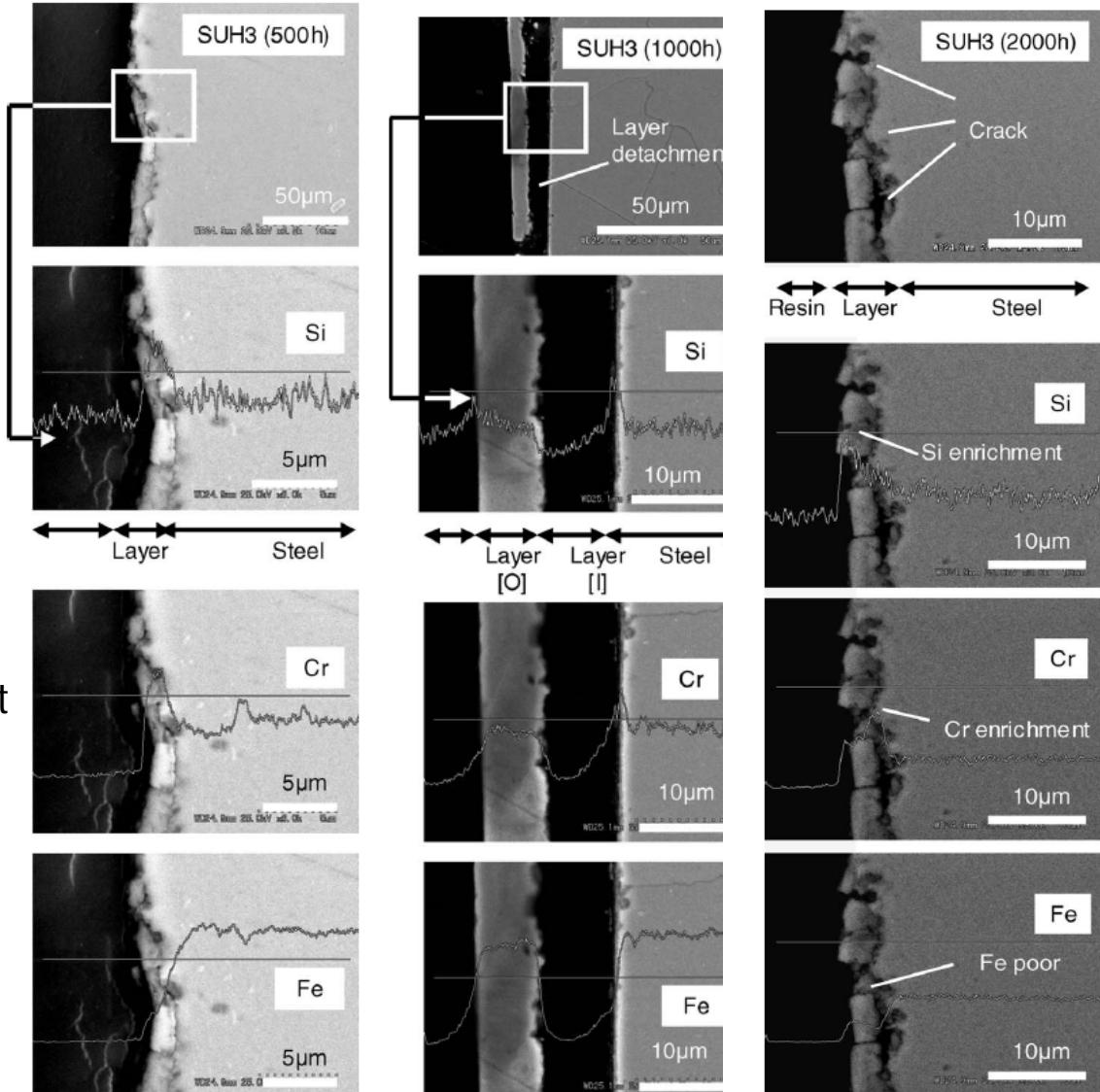
	C	Si	Mn	P	S	Cr	Mo
SUH3	0.42	1.90	0.49	0.023	0.002	10.39	0.72

Test conditions

Exposure time (h)	500	1000	2000	
Flow velocity (m/s)	1	1	1	
Temperature of Pb–Bi (°C)	550	550	550	
Oxygen concentration (wt%)	1.7×10^{-8}	1.7×10^{-8}	1×10^{-6}	
Test steel	SUH3 NTK04L Recloy10	SUH3 NTK04L Recloy10	SUH3 NTK04L Recloy10	

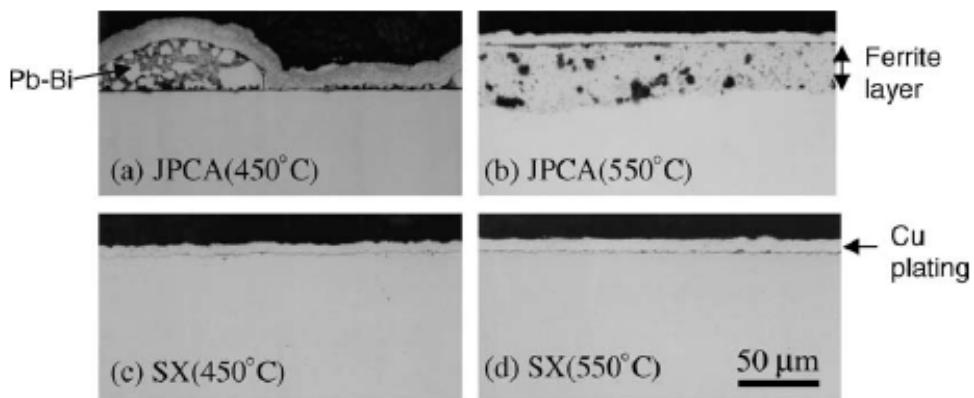
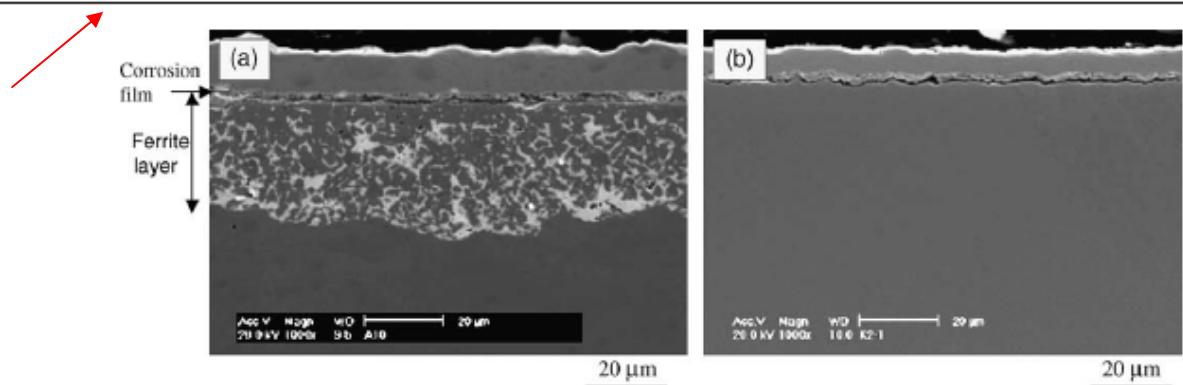
- 2 wt% Si in oxide layer;
- Excellent corrosion resistance in low oxygen LBE;
- Growth, destruction and detachment of oxide layer in high oxygen LBE.

M. Kondo, M. Takahashi, J. Nucl. Mater. 356 (2006) 203.



Austenitic steels

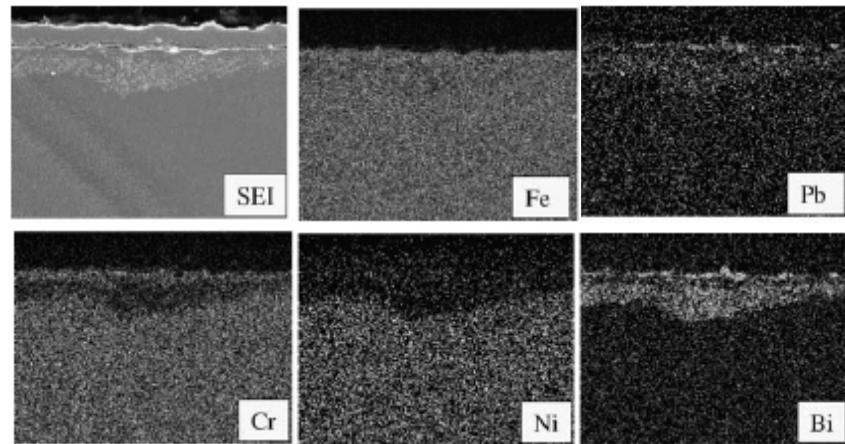
	C	Si	Mn	Cr	Ni	Mo	Fe	Ti	Cu
JPCA	0.058	0.50	1.54	14.14	15.87	2.29	balance	0.22	—
316SS	0.04	0.69	1.22	16.83	10.79	2.06	balance	—	—
SX	0.010	4.80	0.60	17.58	19.08	0.356	balance	—	2.14



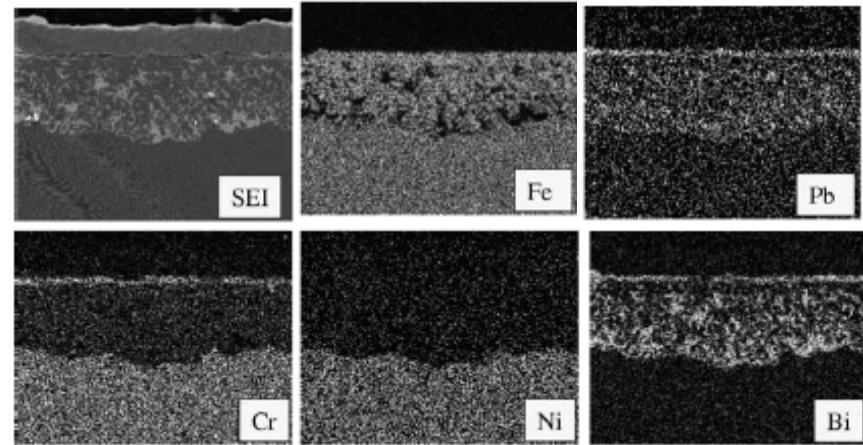
Y. Kurata, M. Futakawa, J. Nucl. Mater. 325 (2004) 217.

Austenitic steels

316



JPCA



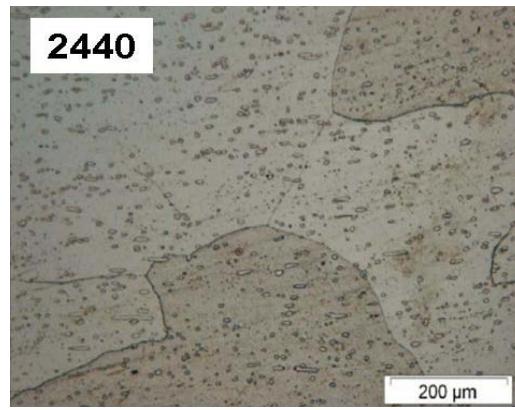
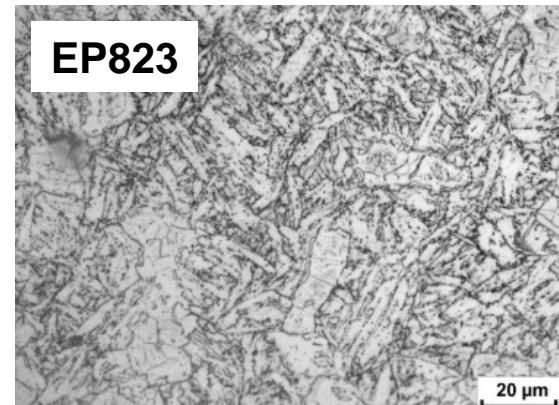
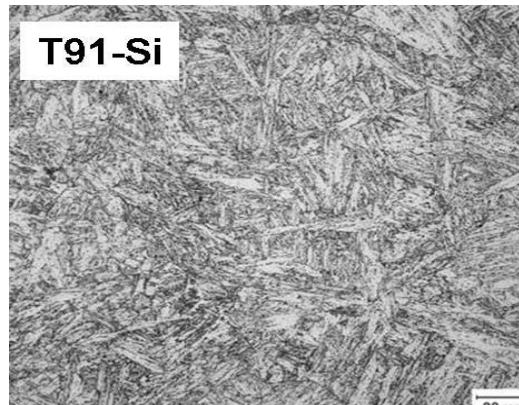
- Protective oxide layer on all three steels at 450° C
- Dissolution of Ni, Cr underneath oxide layer in 316 and JPCA;
- No dissolution of Ni, Cr at 550° C in SX;
- **Oxide film of Si, O protects from dissolution.**

Materials

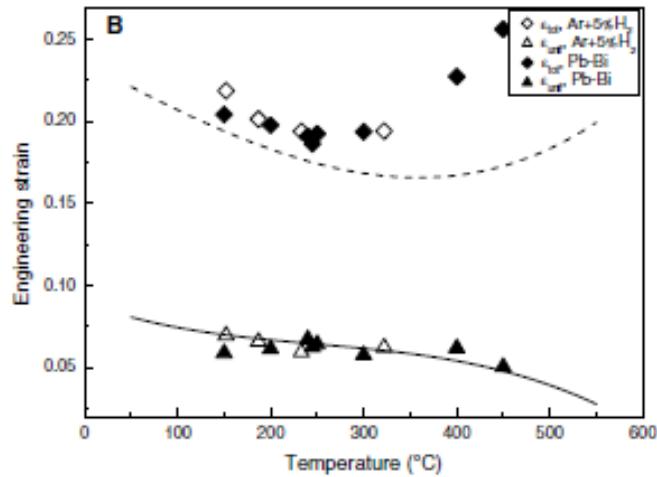
- T91 (standard) - ferritic-martensitic
- T91-Si - ferritic-martensitic
- EP-823 (standard) - ferritic-martensitic
- Heat 2439 - bainitic
- Heat 2440 - ferritic

	C	Cr	Ni	Si	Mo	Mn	Al	V	W	Nb	P	S	N
T91	0.1	8.99	0.11	0.22	0.89	0.38	0.01	0.21	-	0.06	0.02	0.004	0.044
T91-Si	0.13	8.10	0.99	1.45	0.86	0.52	0.003	0.20	-	0.074			0.054
EP-823	0.16	11.70	0.66	1.09	0.74	0.55	-	0.30	0.60		0.014	0.004	
2439	0.19	11.58	0.48	2.75	1.03	0.37	0.042	0.31	0.48		0.004	0.004	
2440	0.19	13.52	0.51	4.83	1.00	0.38	0.043	0.31	0.45		0.004	0.004	

Materials

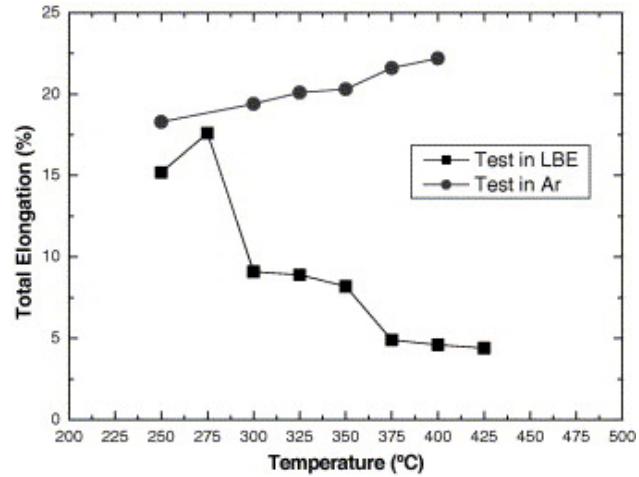


SSRT results: T91

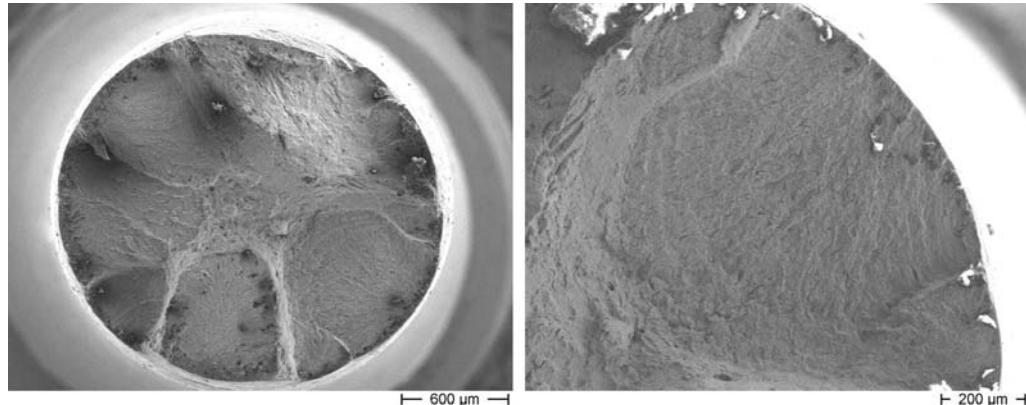


J. Van den Bosch et al., J. Nucl. Mater. 356 (2006) 237

VS.



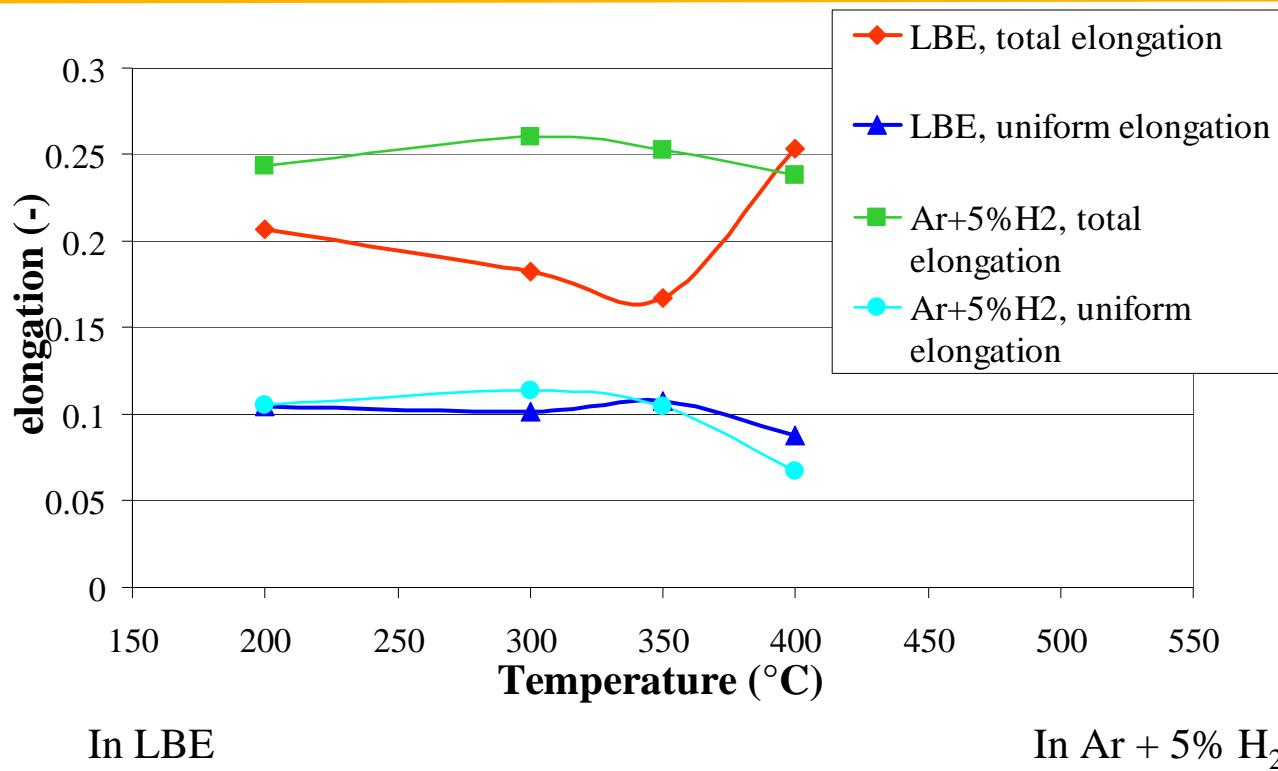
Y. Dai et al., J. Nucl. Mater. 356 (2006) 222



Ductility trough 200-425° C

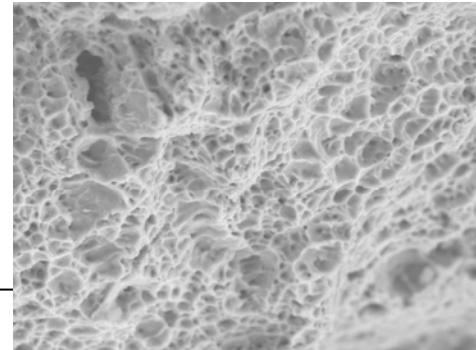
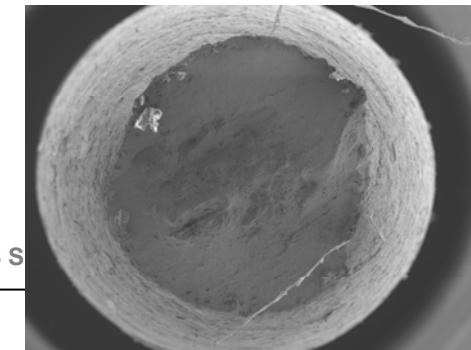
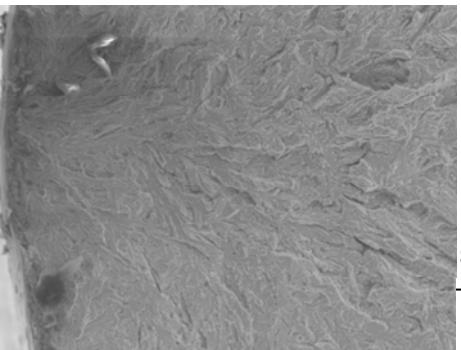
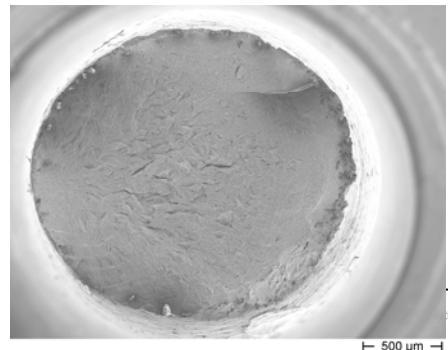
- Presence of microcracks;
- Use of flux to improve wetting.

SSRT results: T91-Si

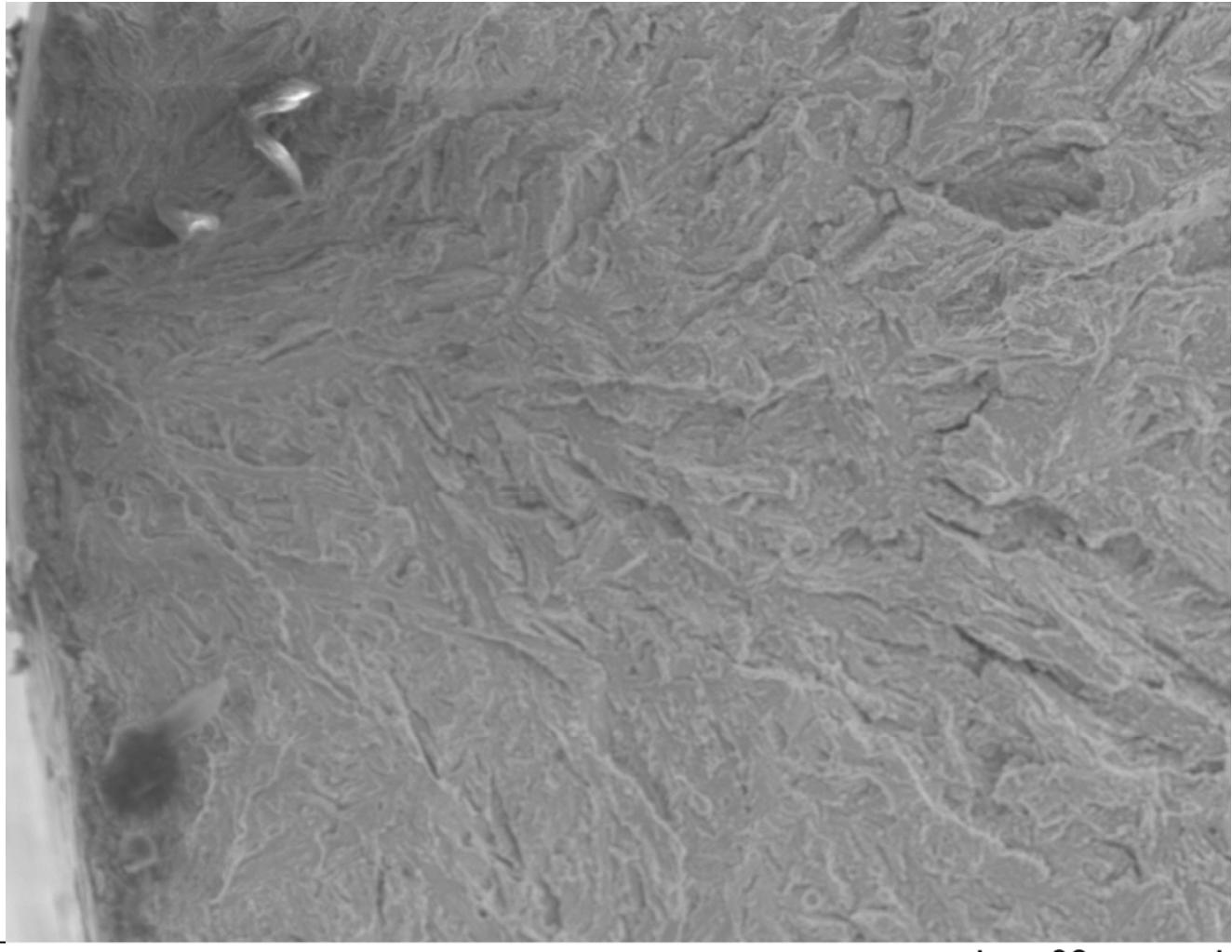
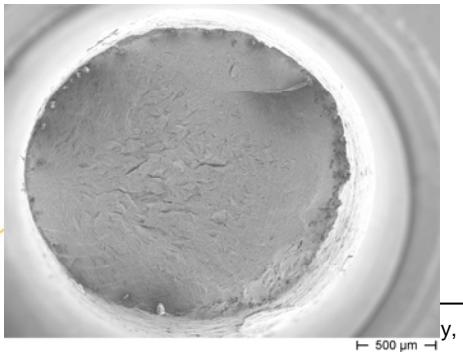


In LBE

In Ar + 5% H₂

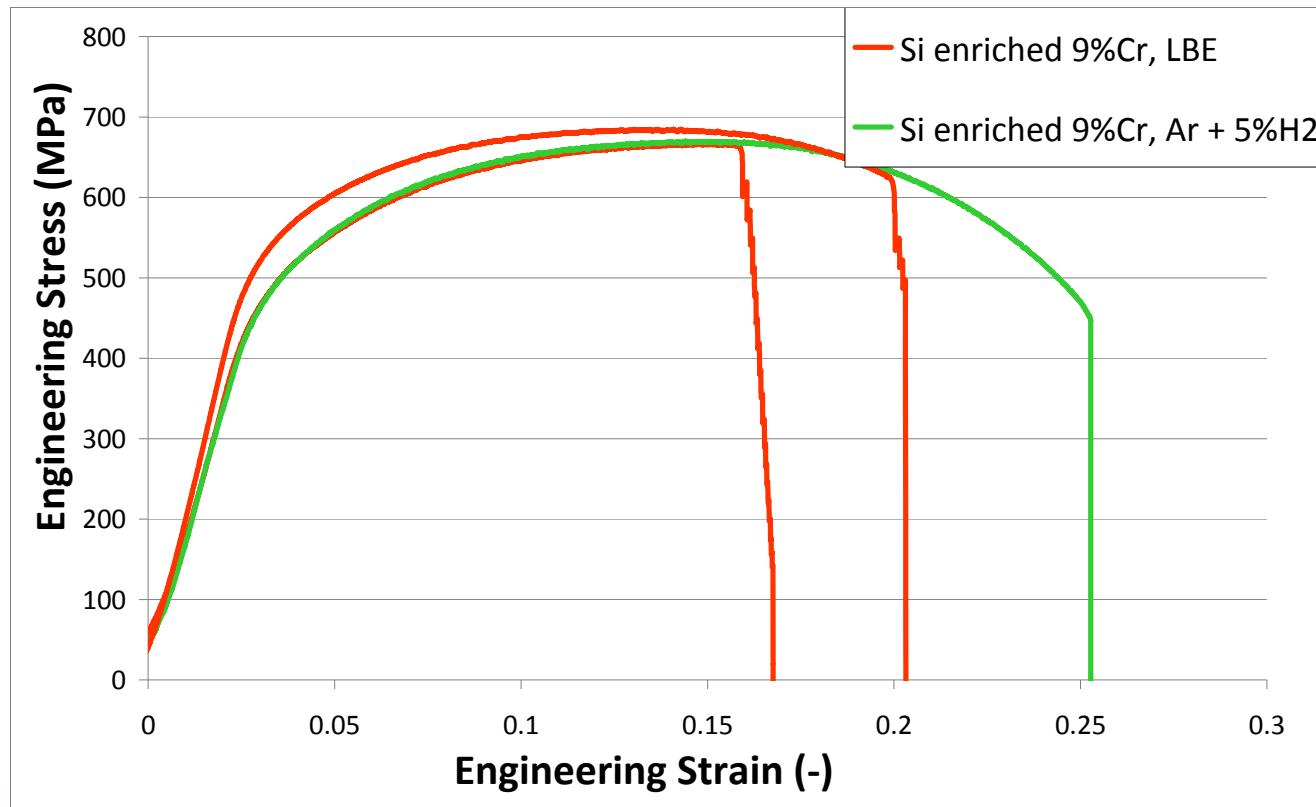


SSRT results: T91-Si

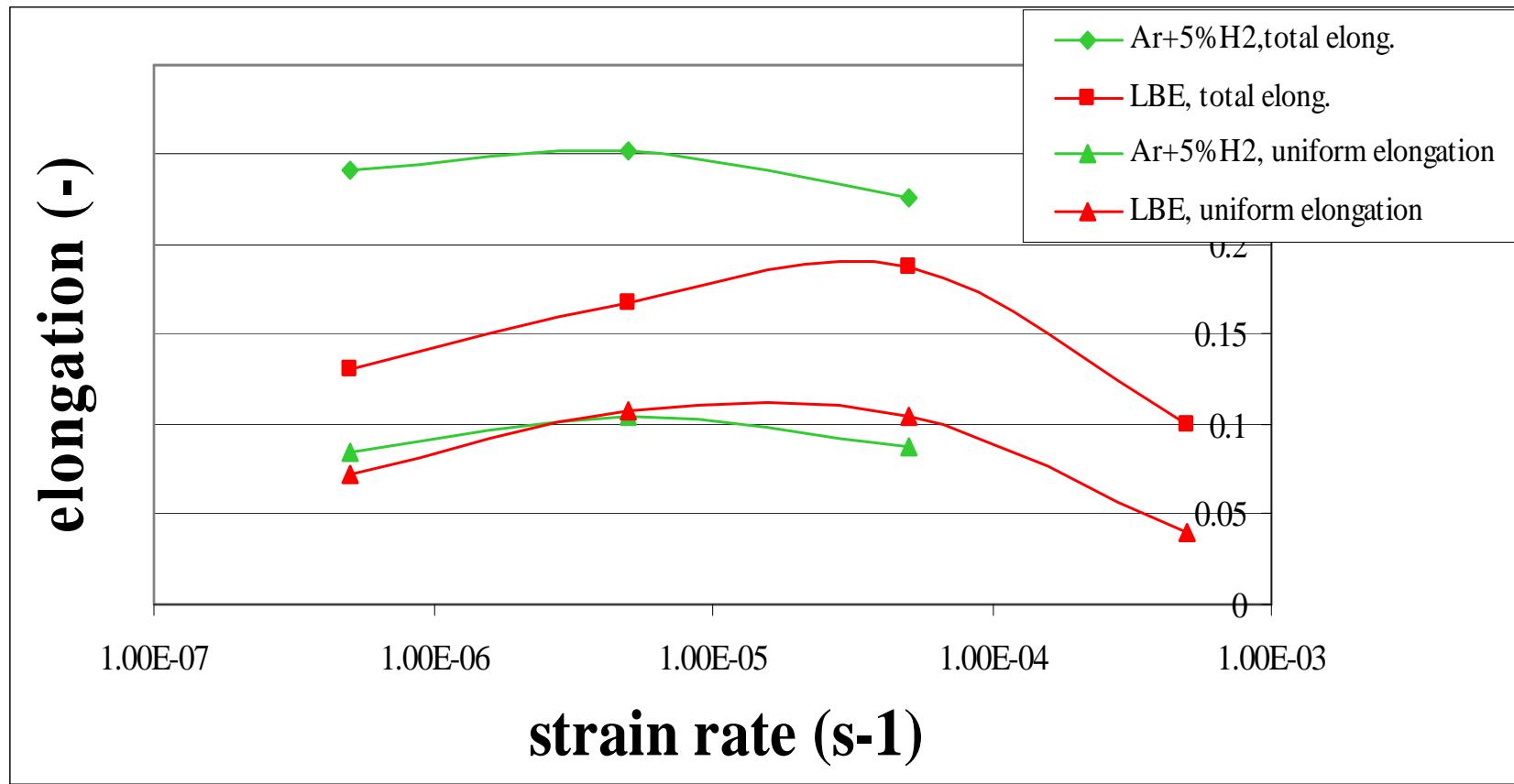


SSRT results: T91-Si

350 ° C, strain rate of 5×10^{-6}

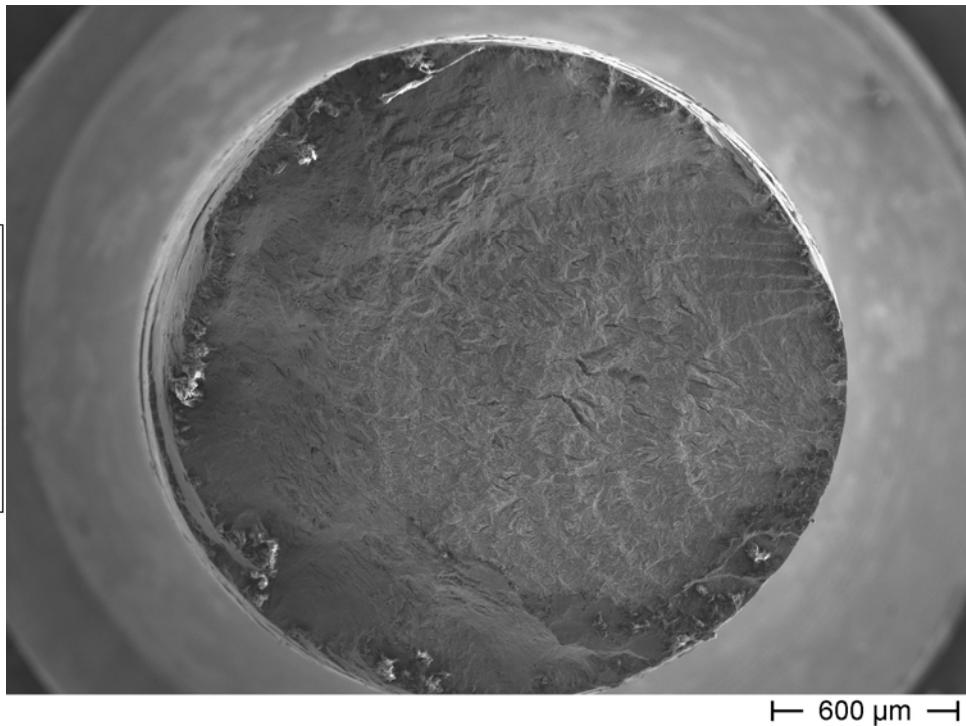
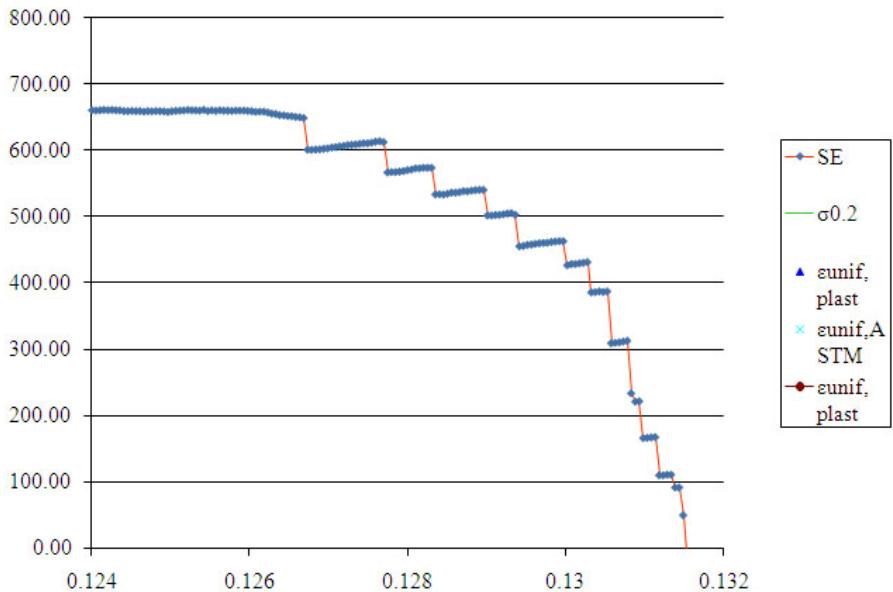


SSRT results: T91-Si

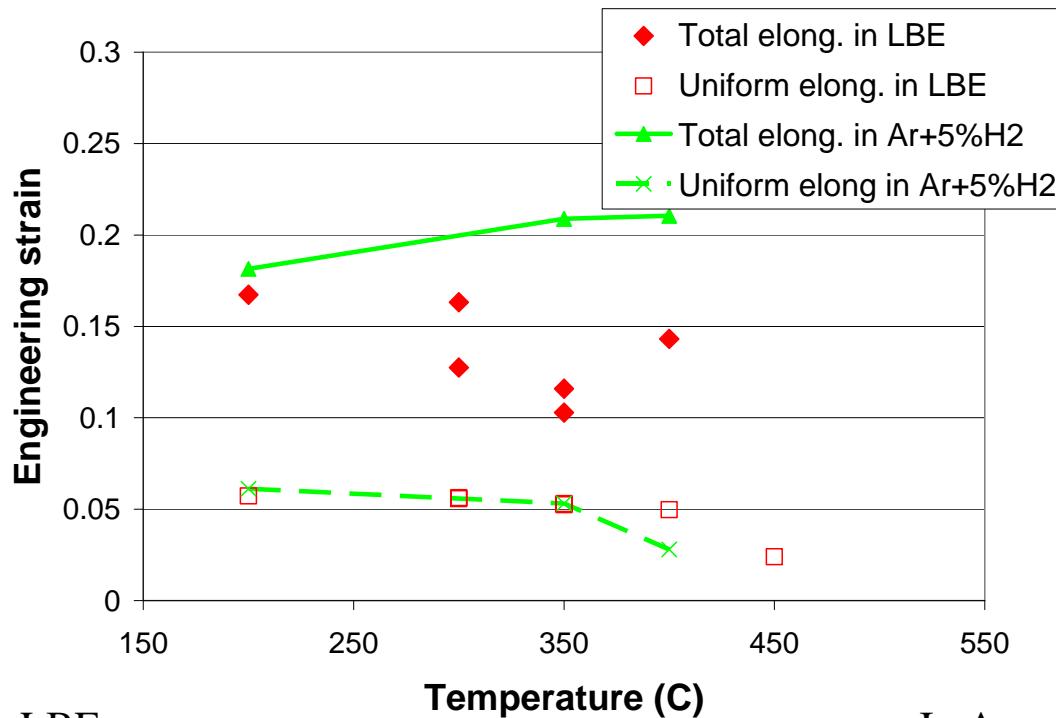


SSRT results: T91-Si

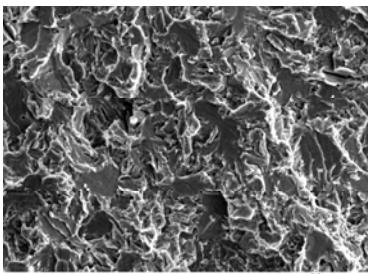
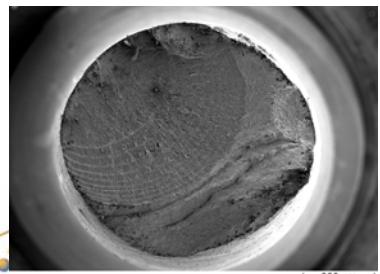
350 ° C, strain rate of 5×10^{-7}



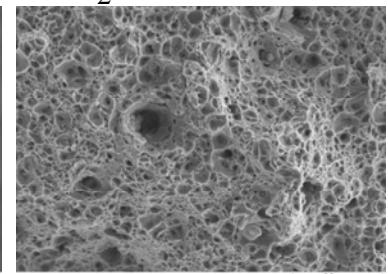
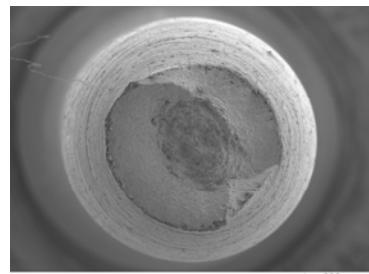
SSRT results EP-823



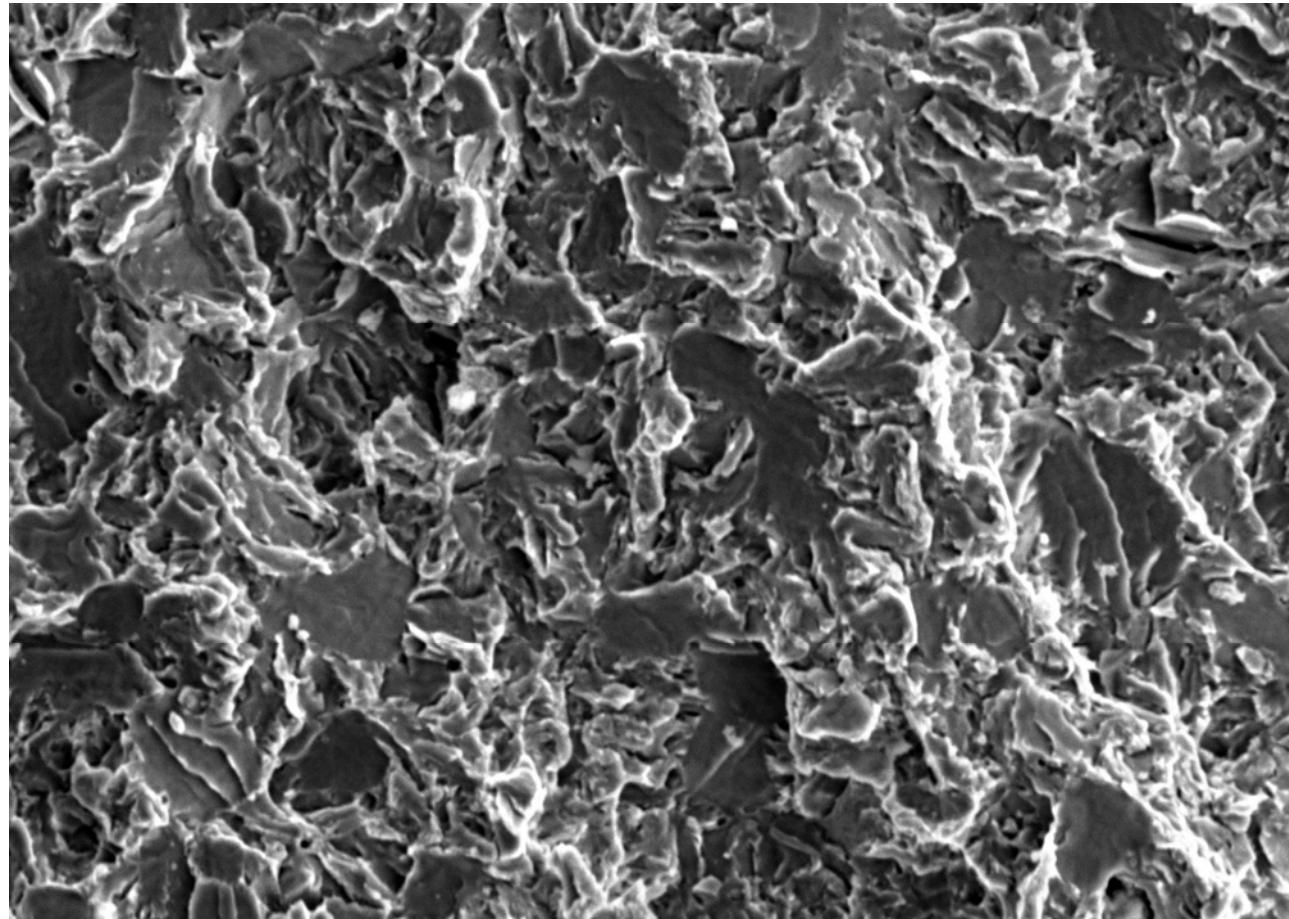
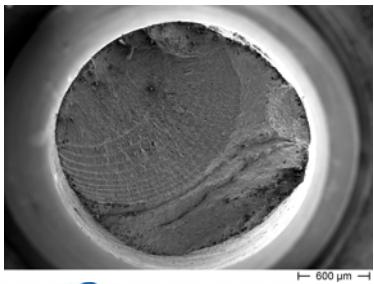
In LBE



In Ar + 5% H₂

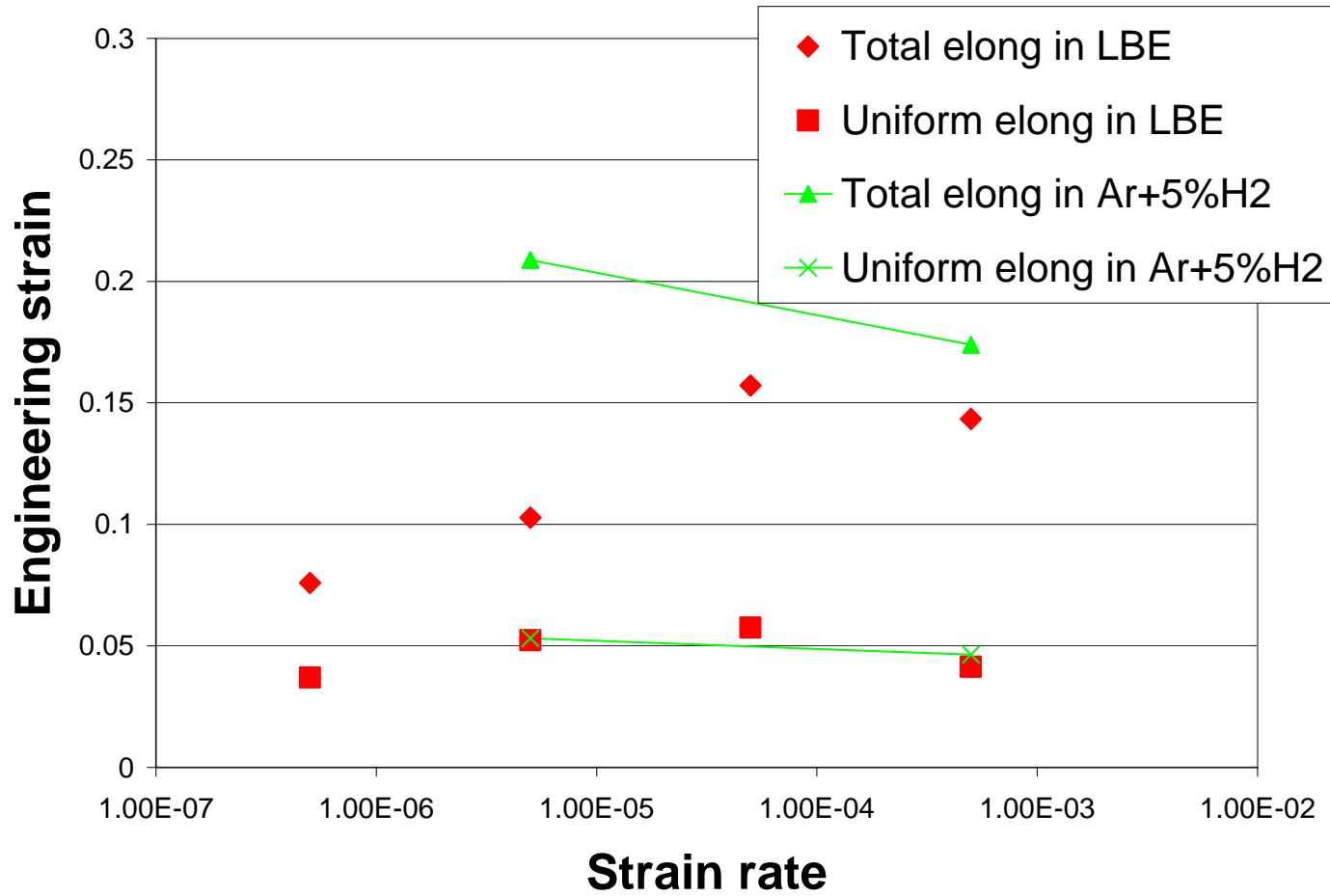


SSRT results EP-823

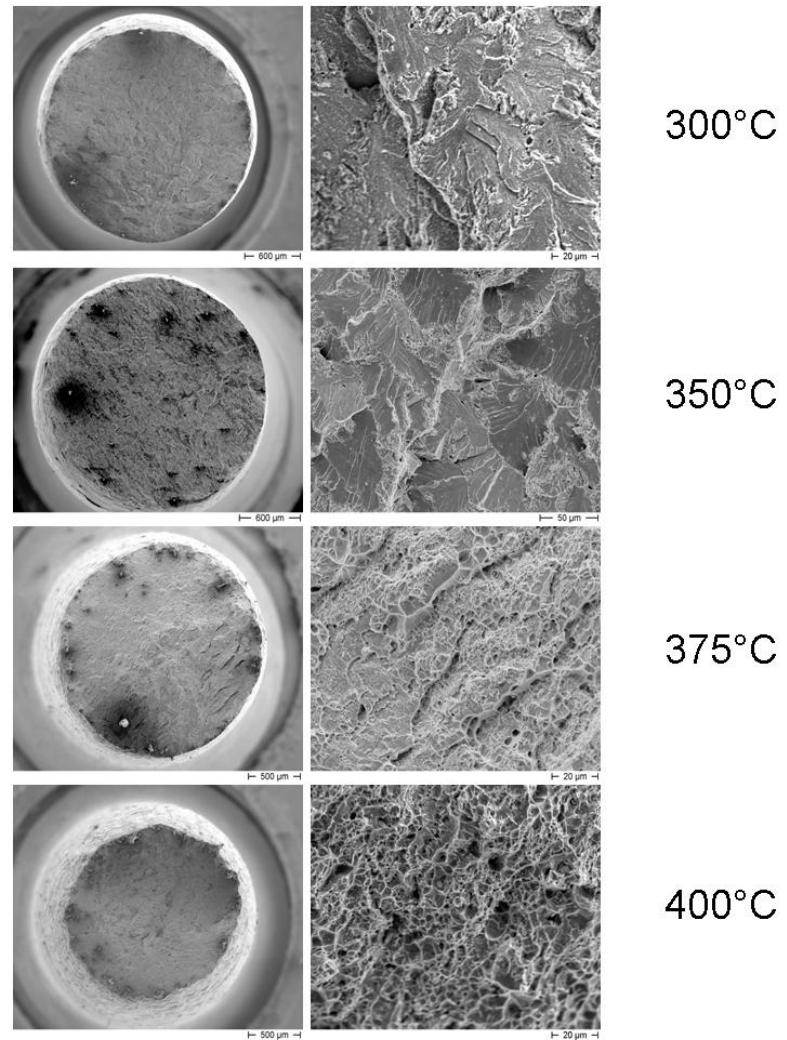
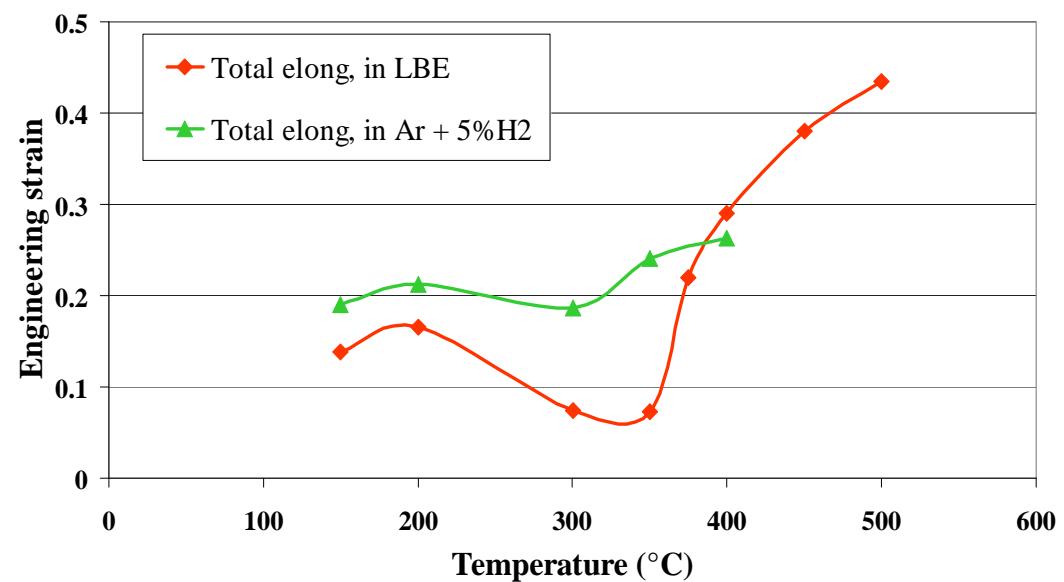


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SSRT results EP-823



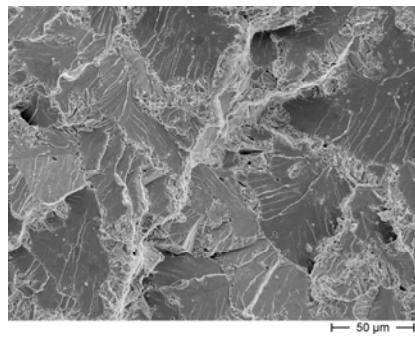
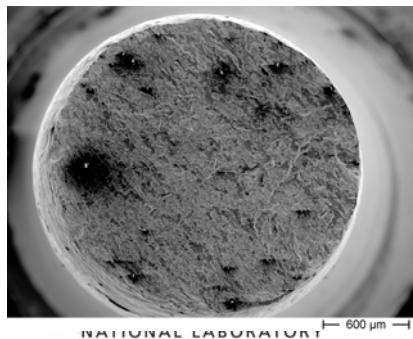
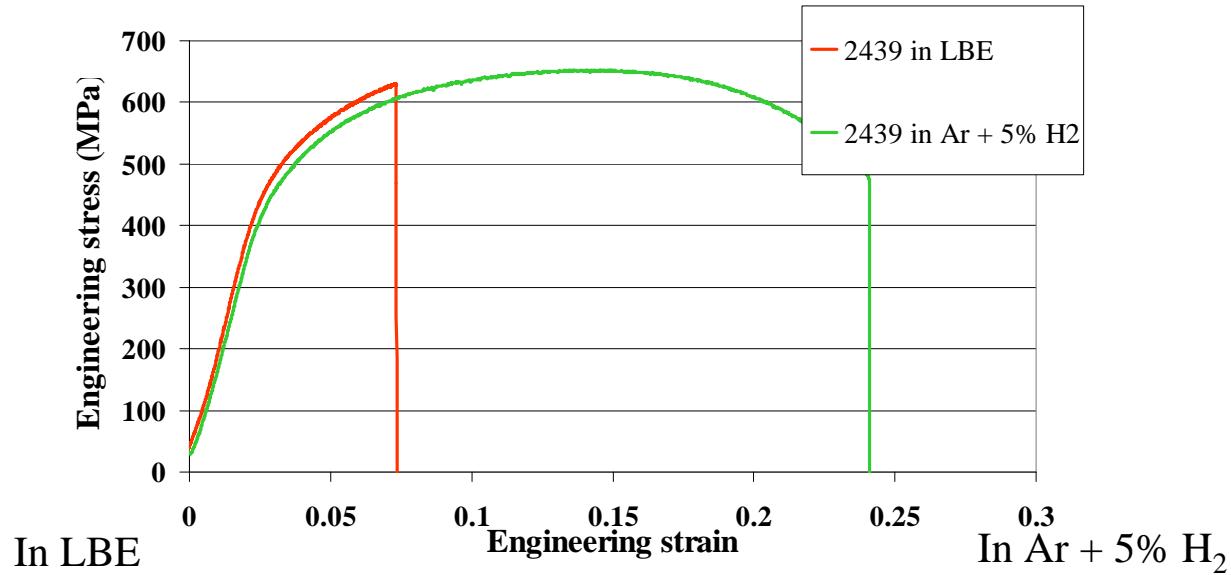
SSRT results: 2439



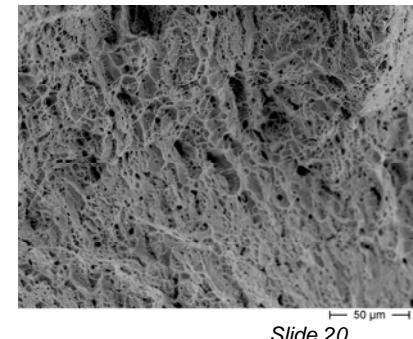
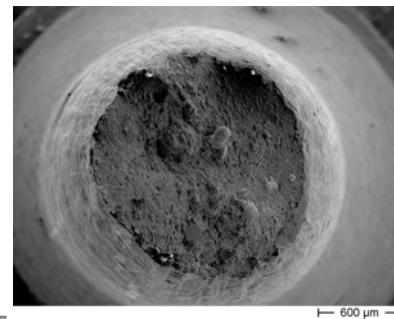
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SSRT results: 2439

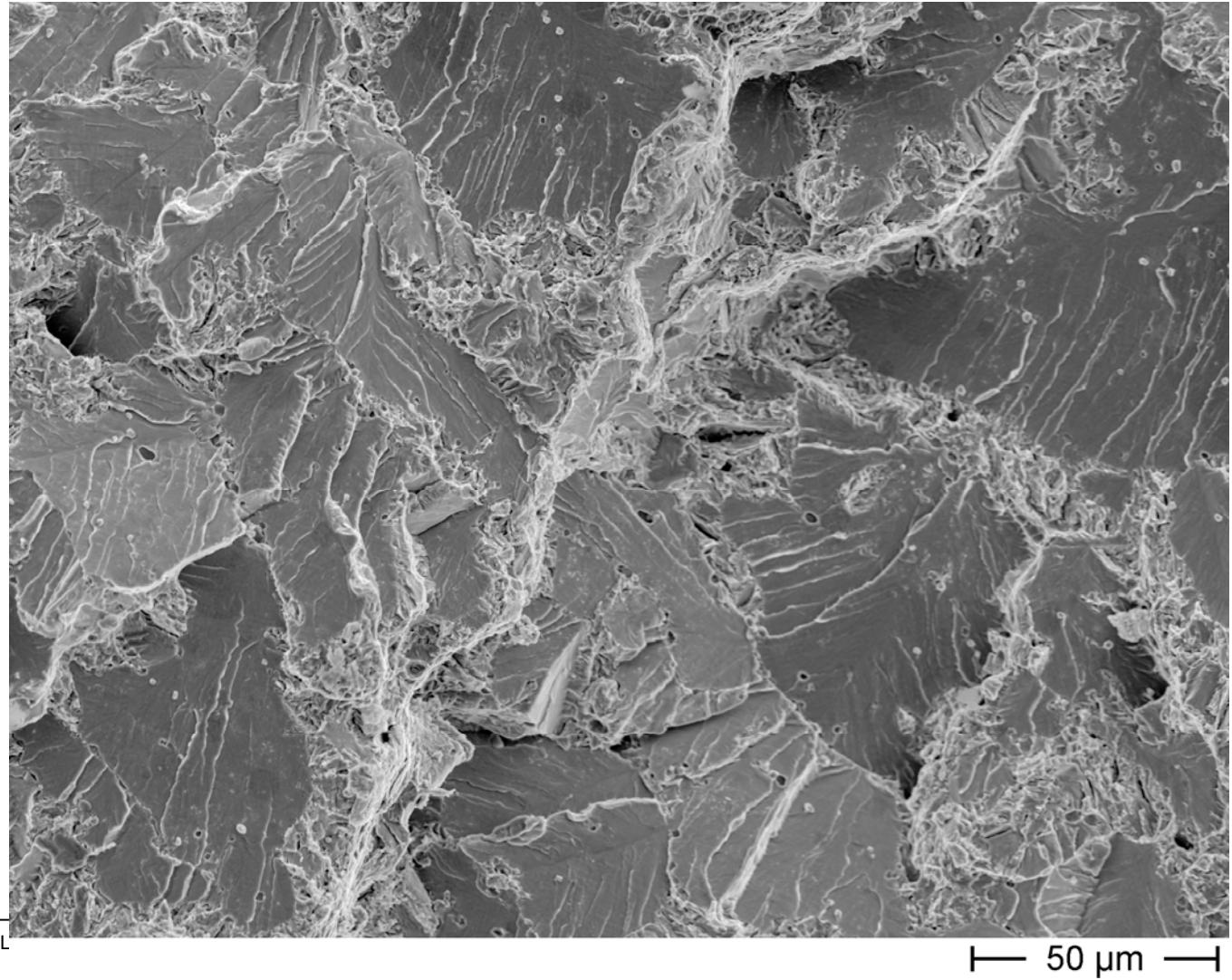
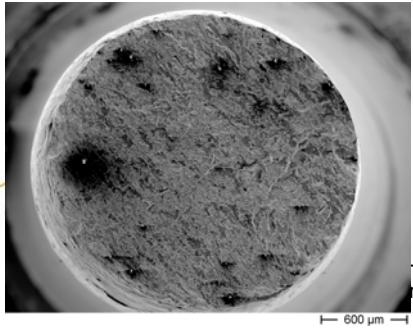
350 ° C, strain rate of 5×10^{-6}



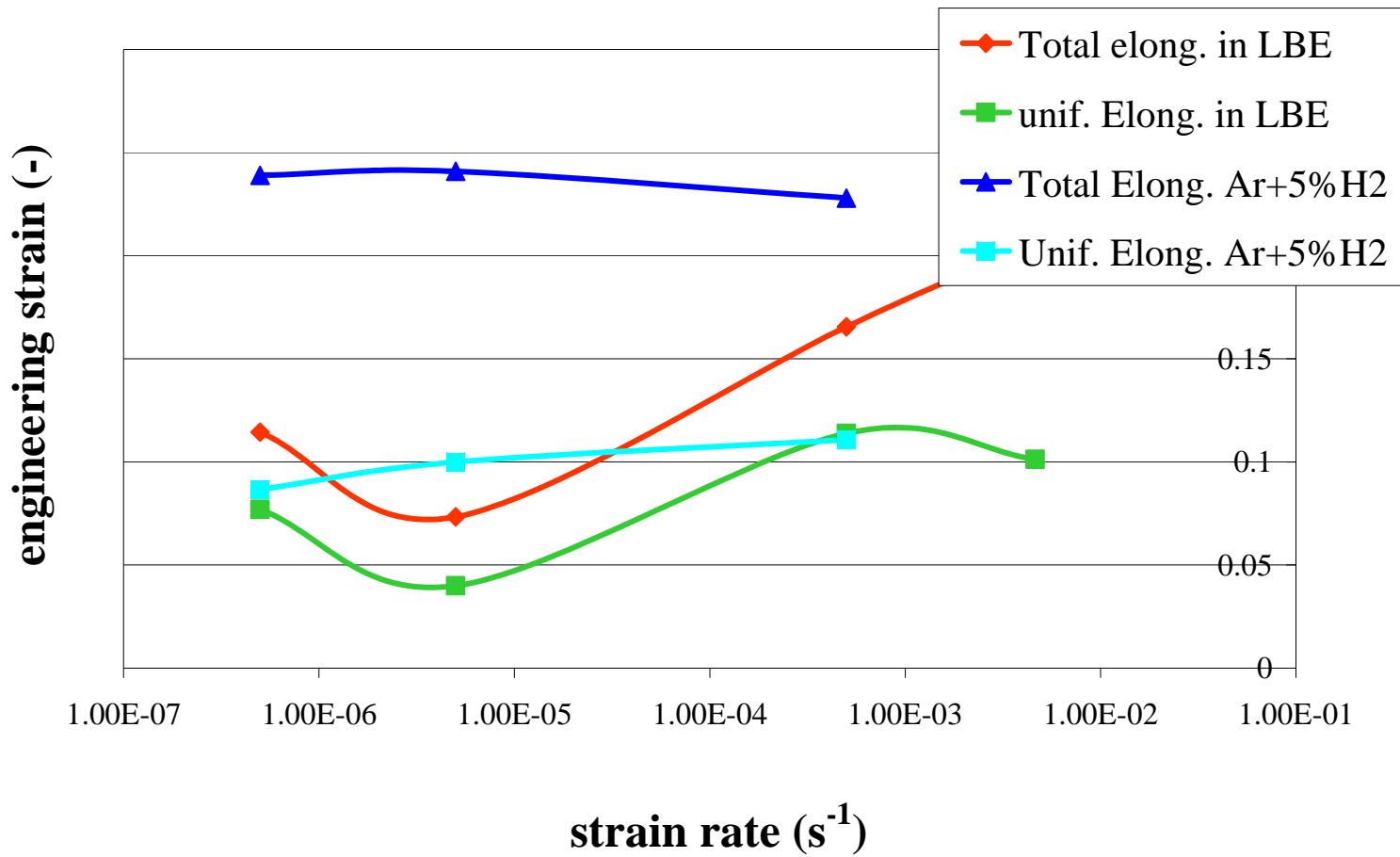
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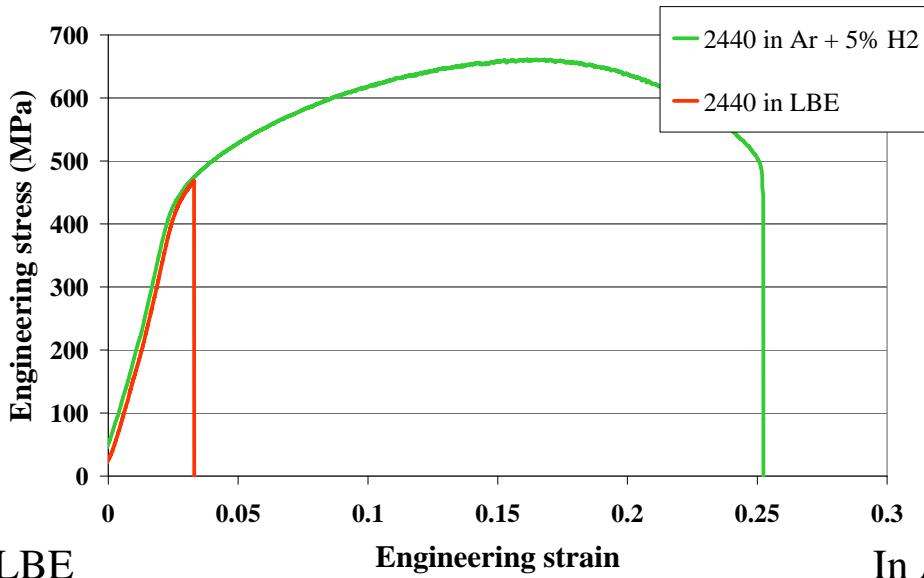
SSRT results: 2439



SSRT results 2439



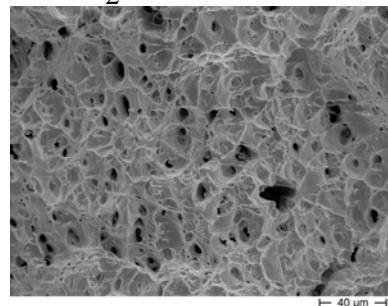
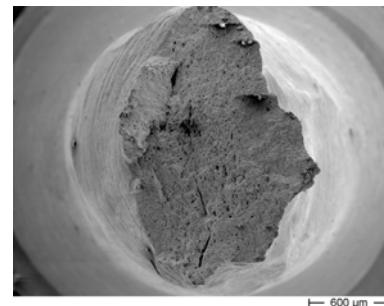
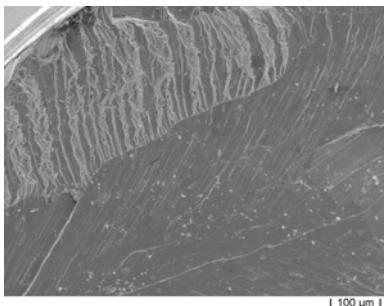
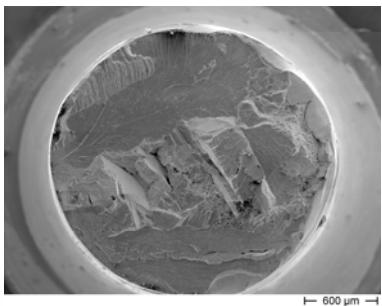
SSRT results: 2440



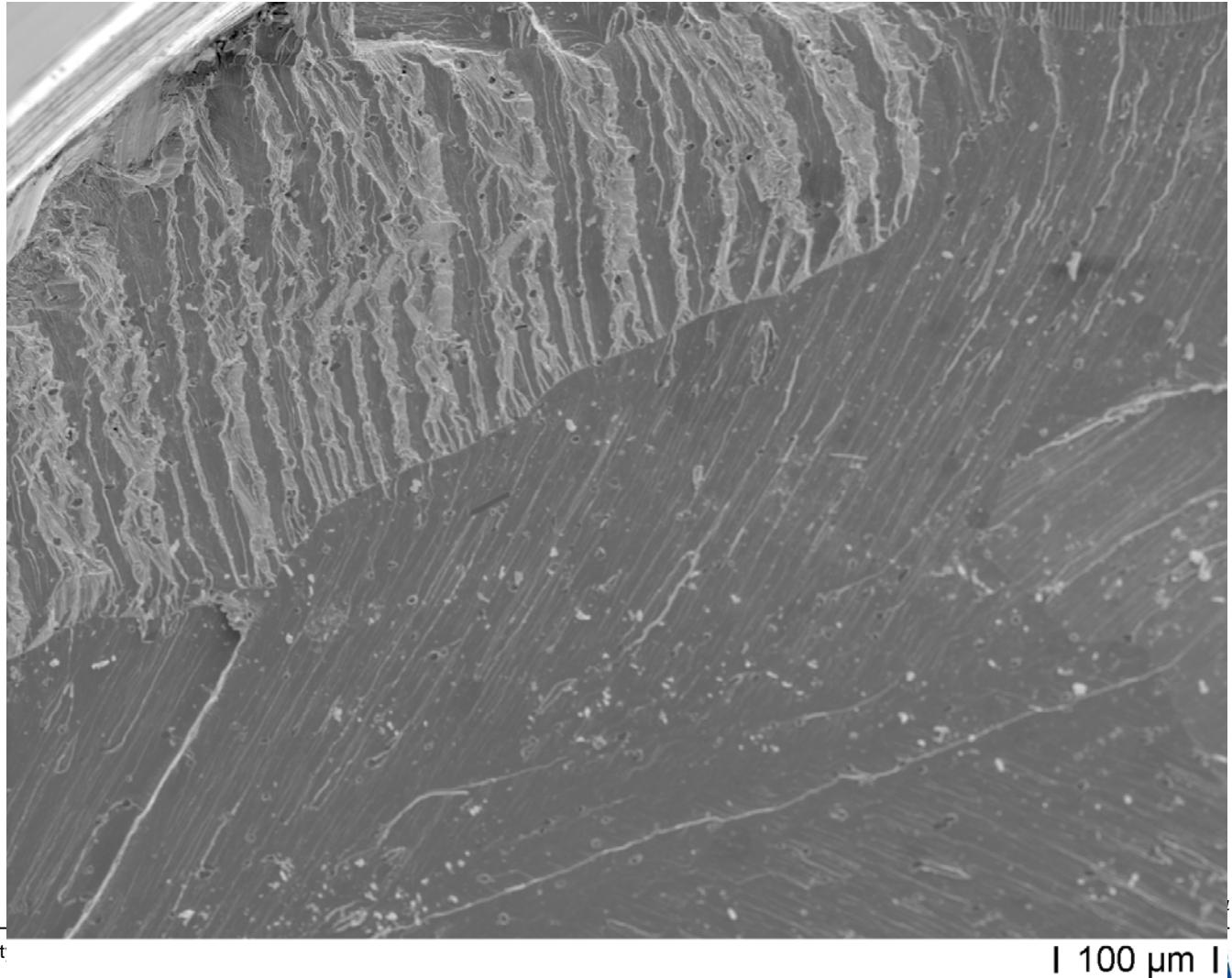
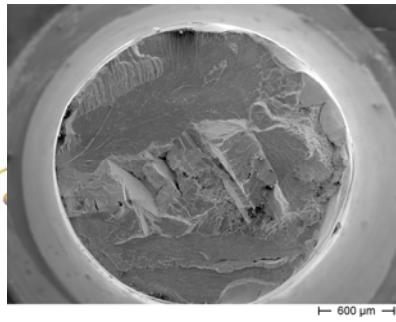
In LBE

Engineering strain

In Ar + 5% H₂



SSRT results: 2440



Focusing thoughts.....

- **Elevated Si steels have better corrosion resistance in LBE;**
- **For cylindrical samples without surface cracks or use of chemical flux, LME occurs more reproducible in elevated Si steels;**
- **More severe LME in high Si steels when it occurs?**
 - Difficult to say for T91-Si and EP-823 vs T91 due to large spread in T91 results
 - 2439 and 2440 (>2 wt% Si) show severe deterioration in total elongation
 - > Probable grain size effect (importance of small grains!)

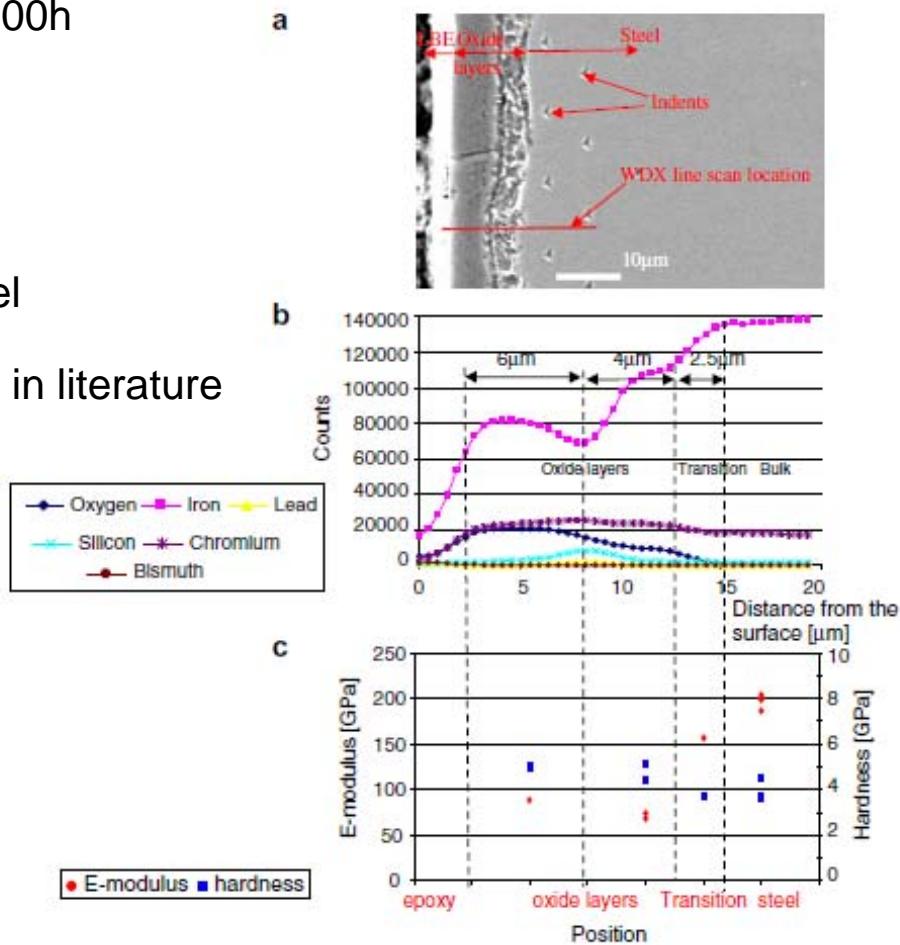
Influence of oxide layer morphology and strength on LME initiation?

- Focus on T91 vs EP823

T91

T91 exposed to flowing LBE at 535° C for 600h

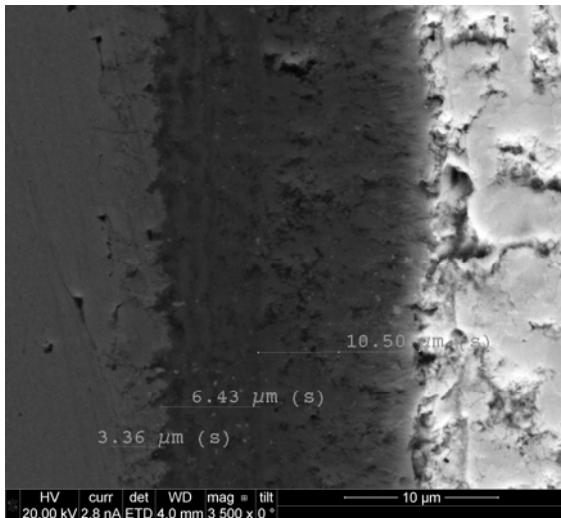
- 10^{-6} wt% oxygen in the LBE
- Use of low load nano-indenter
- Lower E-modulus of oxide than bulk steel
- Lower E-modulus of oxide than reported in literature



T91

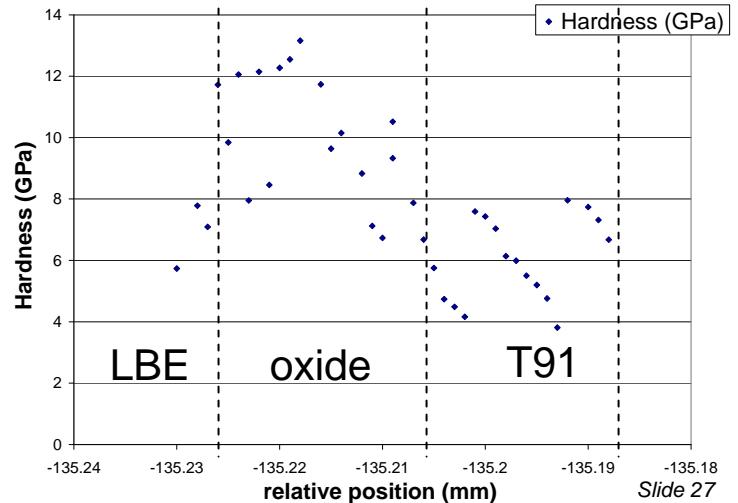
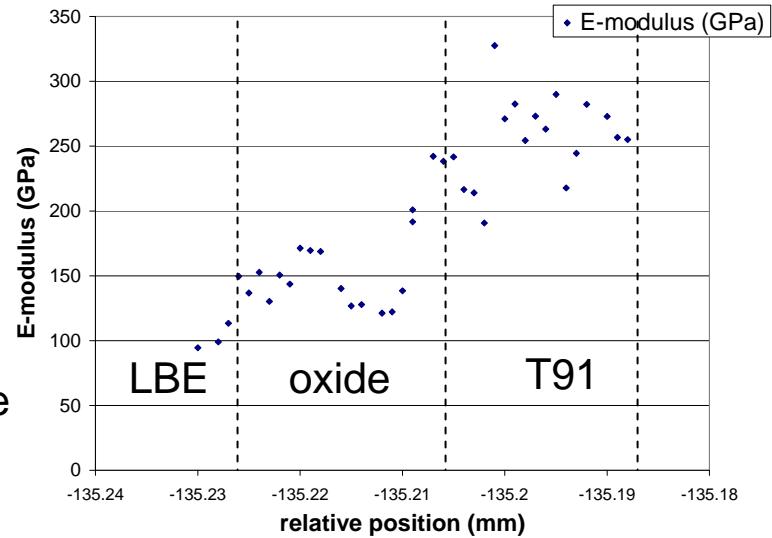
T91 exposed to stagnant LBE at 550° C for 1500h

- oxygen saturated LBE
- Use of high load nano-indenter
- Lower E-modulus of oxide than bulk steel
- Lower E-modulus of oxide than reported in literature

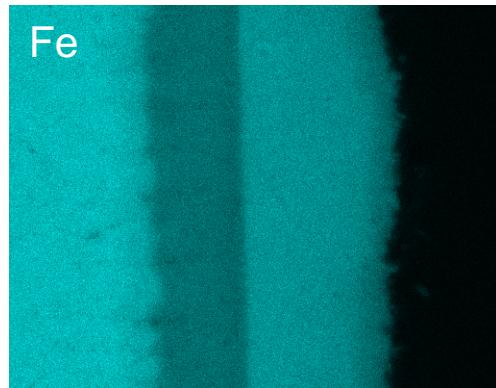
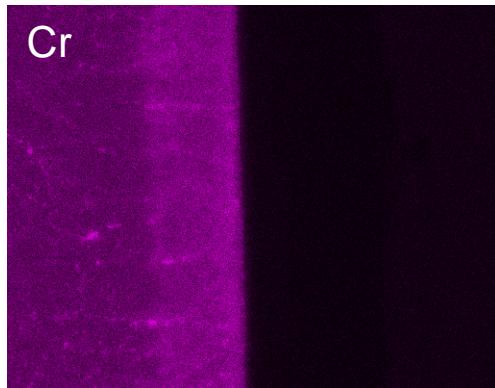
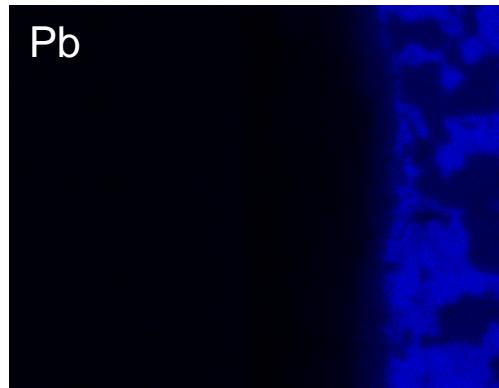
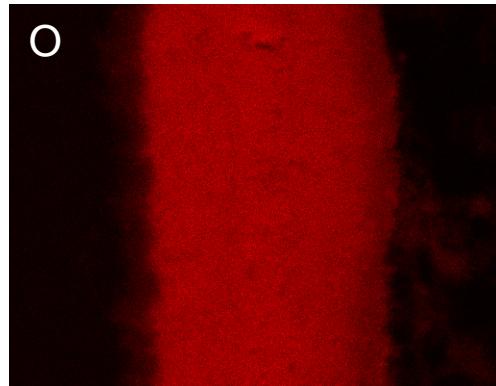
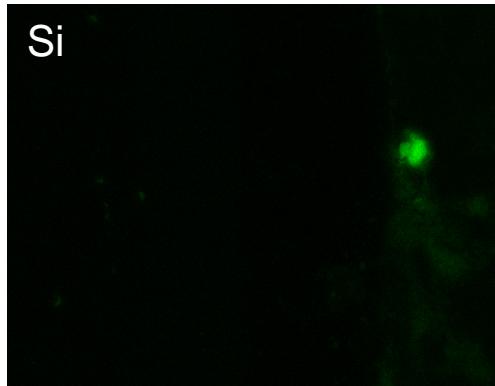
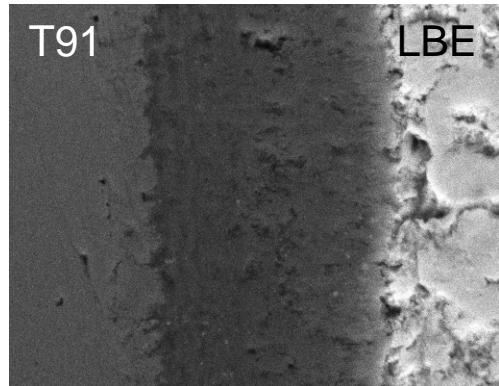


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Operated by Los Alamos National Security, LLC for NNSA



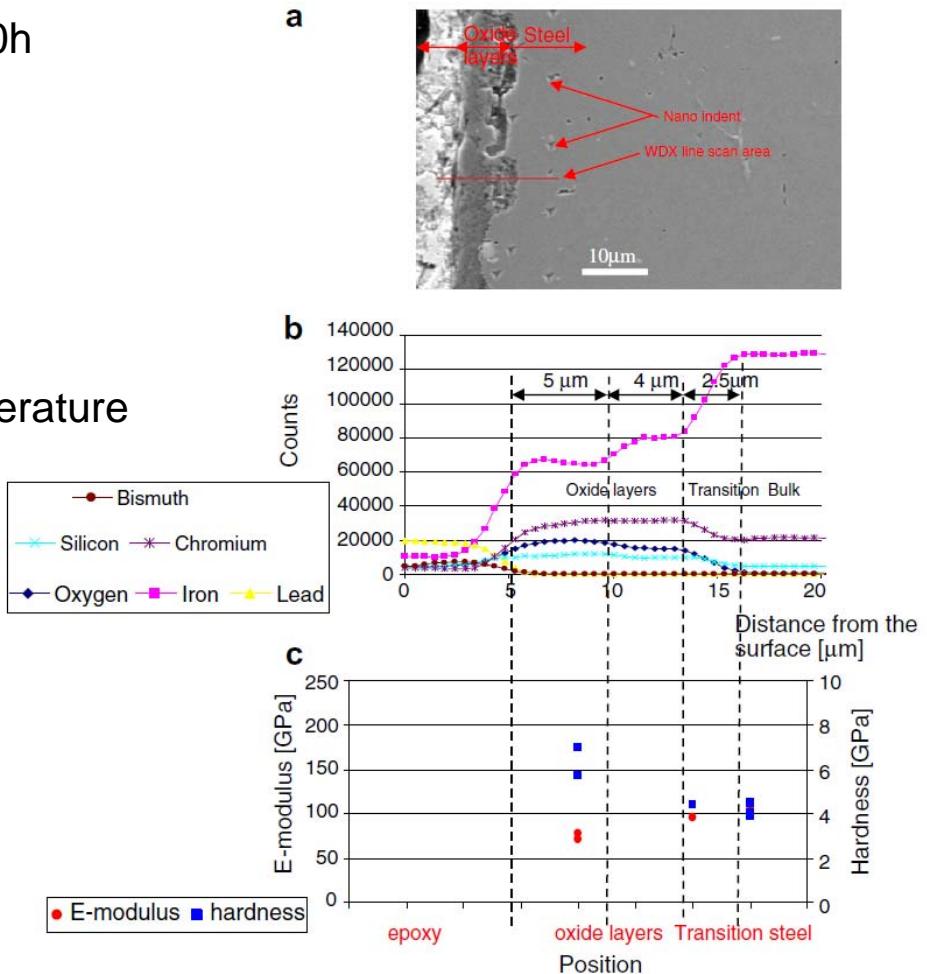
T91



EP823

EP823 exposed to flowing LBE at 535° C for 600h

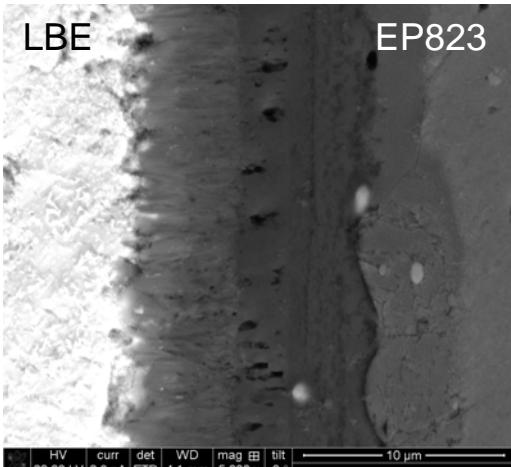
- 10^{-6} wt% oxygen in the LBE
- Use of low load nano-indenter
- Lower E-modulus of oxide than bulk steel
- Lower E-modulus of oxide than reported in literature
- Si enrichment between inner and outer oxide



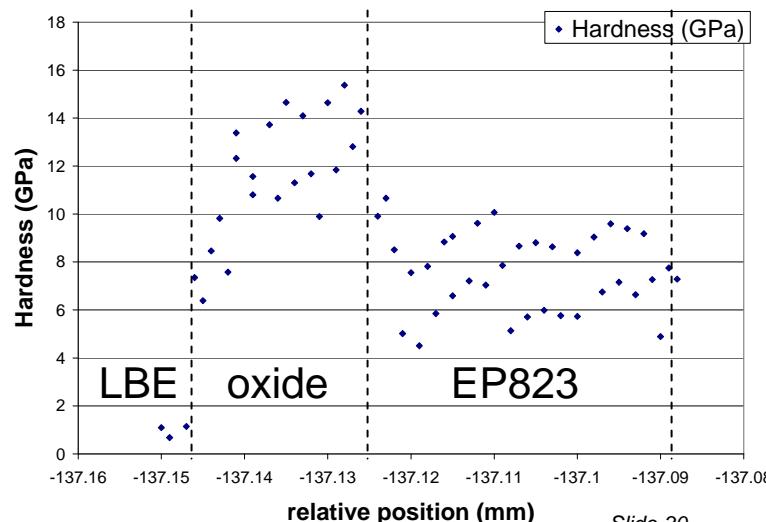
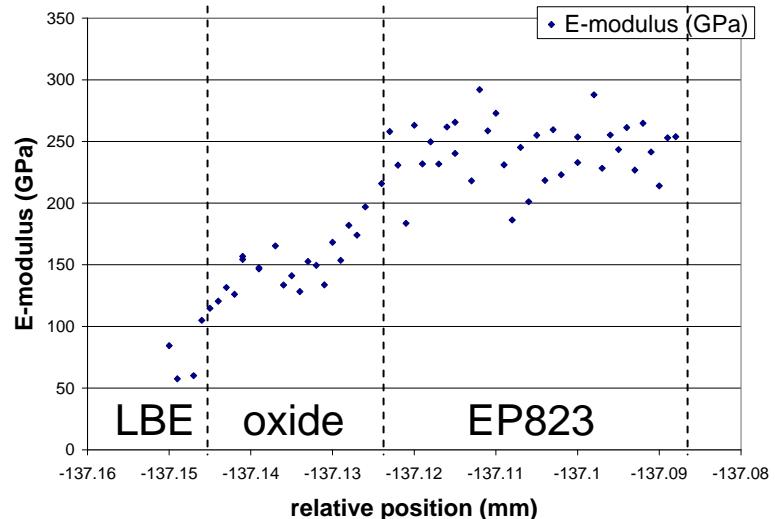
EP823

EP823 exposed to stagnant LBE at 550° C for 1500h

- oxygen saturated LBE
- Use of high load nano-indenter
- Lower E-modulus of oxide than bulk steel
- Lower E-modulus of oxide than reported in literature
- Harder and stiffer oxide layer than T91

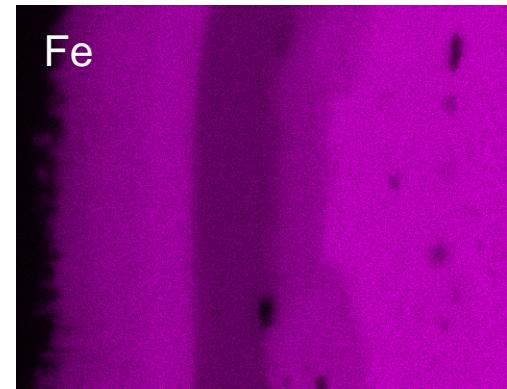
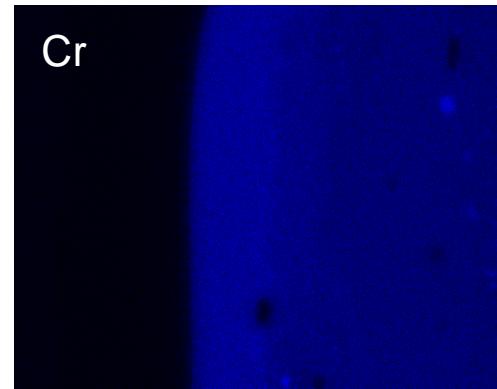
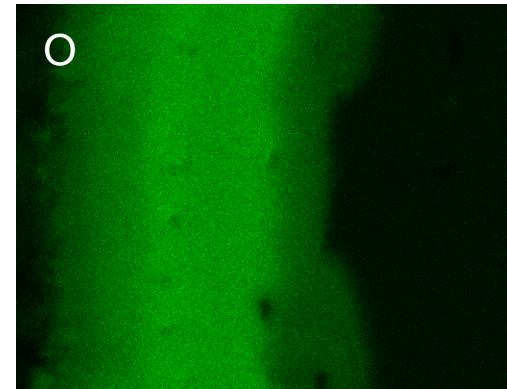
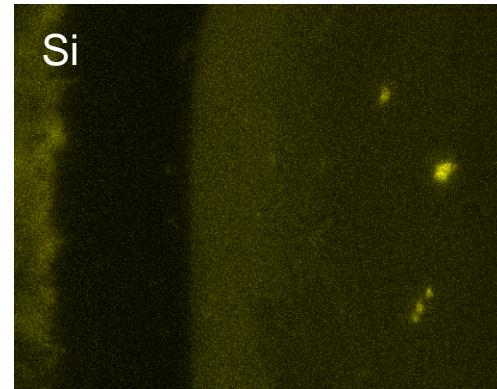
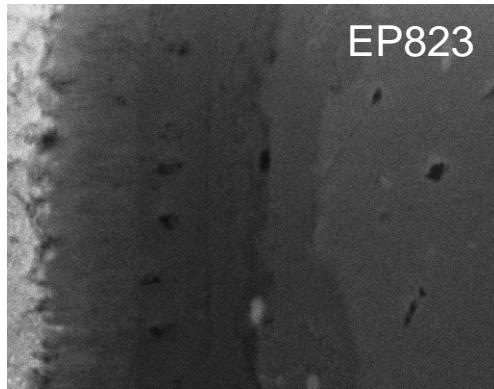


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Slide 30

EP823



Conclusions

- Si addition increases corrosion resistance in LBE but also strongly increases susceptibility to LME;
- Grain size seems to strongly determine severity of decrease in total elongation when LME occurs;
- Nano-indentation shows lower E-modulus of LBE grown oxides on T91 and EP823 than that of the bulk steels;
- EP823 oxide layer is harder and stiffer than that of T91.

Outlook

- SEM examination of oxide layer indents;
- Nano-indentation in cross section as function of temperature;
(Change of oxide layer properties in ductility trough?)
- Micro-pillar testing of separate oxide layer components;
(Which really is the weakest link of the complex oxide?)