Effects of oxygen concentration, strain rate and temperature on LME susceptibility of T91 steel in LBE under low cycle fatigue

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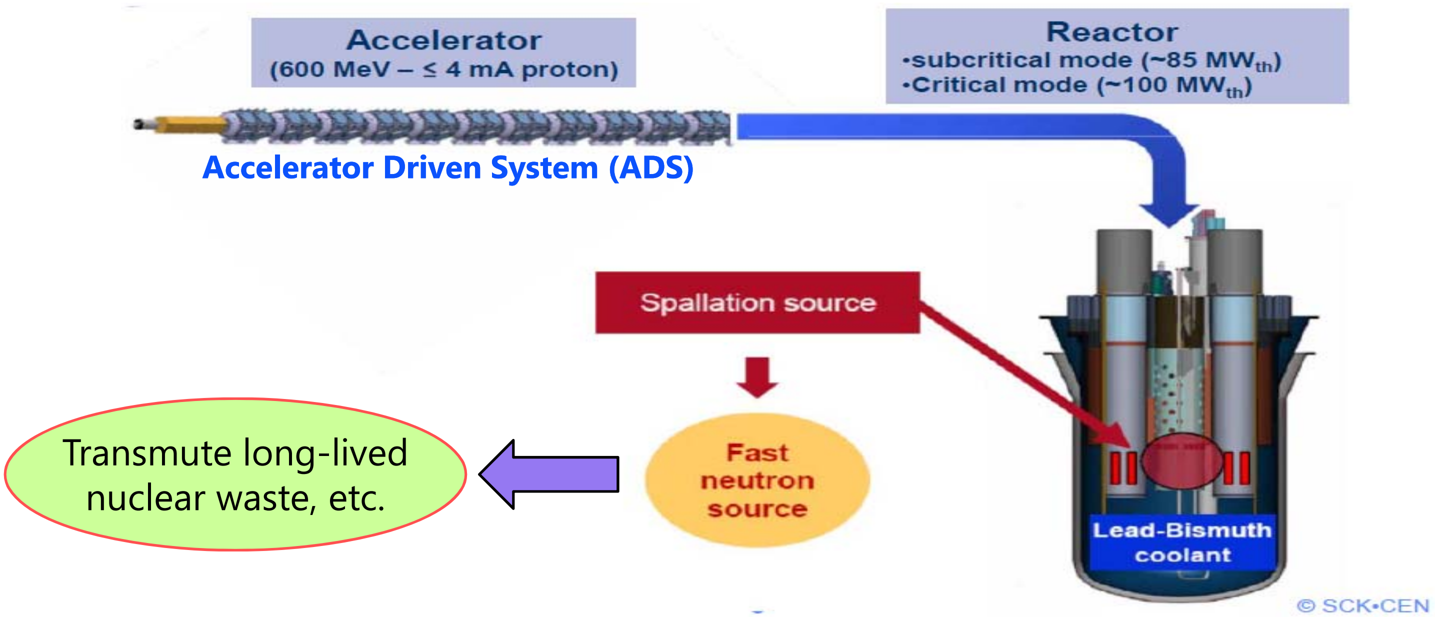
  - Strain rate effect
- Fatigue crack propagation modes
- Temperature dependence of LME susceptibility
- A proposed new LME mechanism
- Conclusions

MYRRHA Reactor

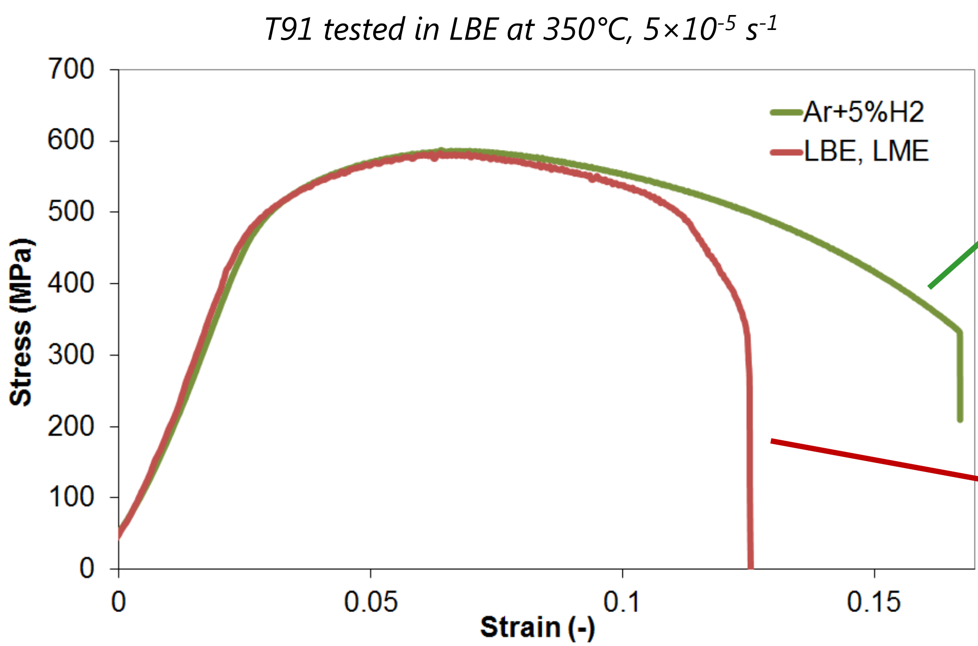


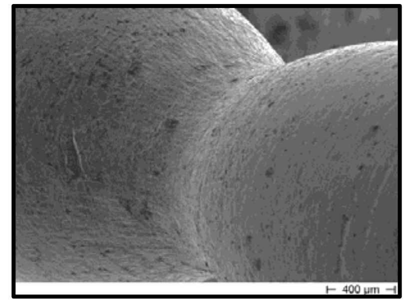
T91 steel is a candidate material for constructing the proton beam window

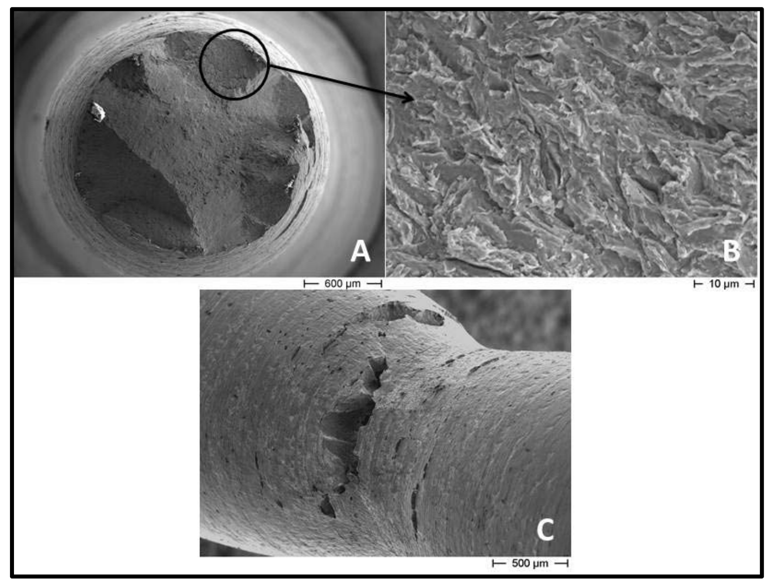
- **Potential fatigue issues**
  - Thermal effects: regular operation of the reactor, proton beam intensity fluctuations, thermal stripping and stratification
  - Mechanical loadings: pump rotation at 4 Hz
  - Lead-bismuth eutectic (LBE) flow induced vibrations

What is Liquid Metal Embrittlement (LME)?

Liquid metal embrittlement (LME) is a phenomenon, in which normally ductile metallic materials undergo losses of ductility and toughness when stressed in a specific liquid metal.



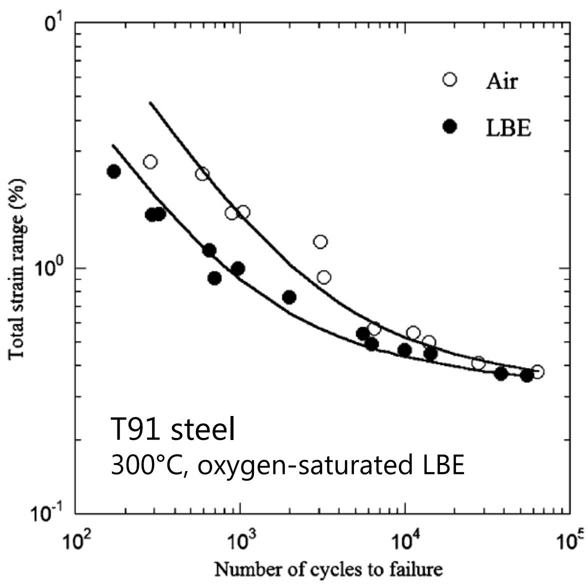




LME results in a decrease of tensile elongation and formation of quasi-cleavage

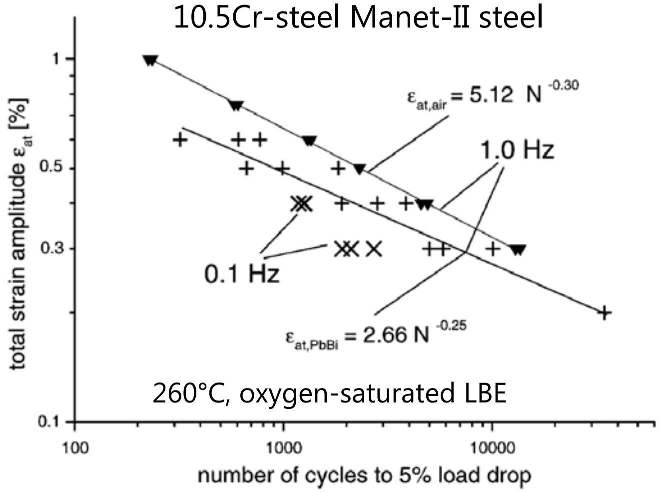
Literature survey

A. Verleene, J.-B. Vogt, 2006



Kalkhof, 2003

10.5Cr-steel Manet-II steel



A. Weisenburger et al, 2008, T91 steel, low cycle fatigue in LBE at 550°C, oxygen concentration = 10^{-6} wt.%

How will LME affect low cycle fatigue behaviour of T91 steel under the conditions as follows?

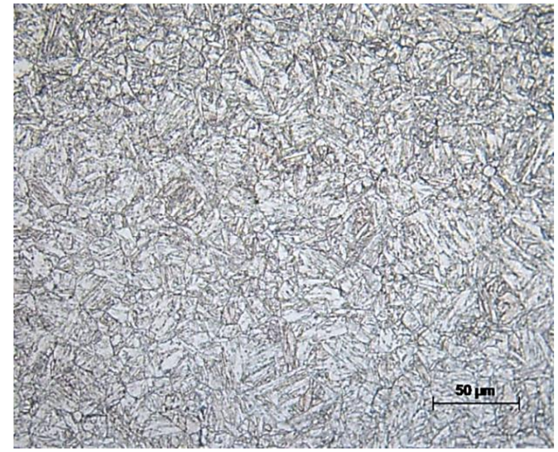
- ✓ Temperature (MYRRHA operation temperature window: 270 to 500°C)
- ✓ Oxygen concentration (MYRRHA operation oxygen regime: 10^{-6} to 10^{-7} wt.%)
- ✓ Strain rates, etc

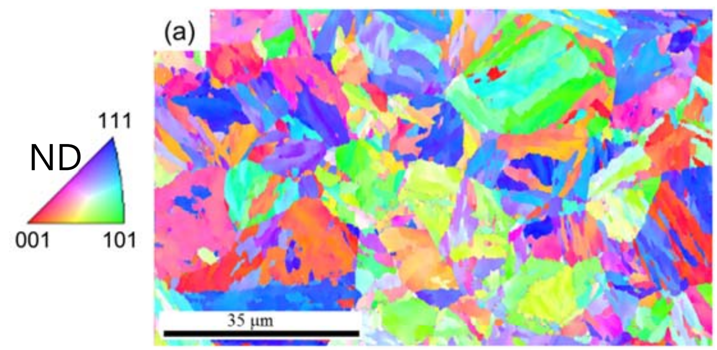
T91 ferritic-martensitic steel

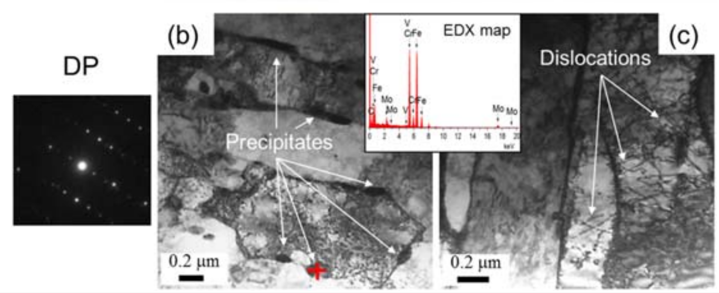
- Heat treatment
  - Austenitized at 1050°C for 15 min and then water quenched, followed by tempering at 770°C for 45 min and then air cooling
- Chemical composition (wt.%)

C	Cr	Ni	Mo	Mn
0.097	8.87	0.12	0.87	0.39
V	**Si**	**Nb**	**N**	**Fe**
0.19	0.22	0.08	0.044	Bal.

- Microstructure
  - Tempered martensitic laths are embedded in the prior-austenite grains
  - The width of the martensitic laths varies from 0.2 to 1.0 μm
  - Carbides are mostly located at lath boundaries
  - Numerous dislocations are distributed in some laths

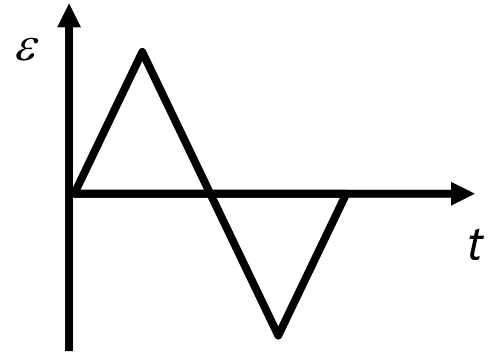


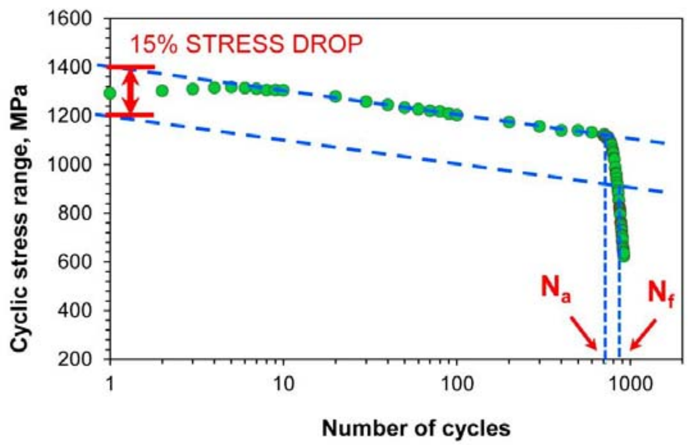




Experimental parameters

- Strain-controlled mode
- Fully pull-push mode with a triangular waveform
- Strain amplitudes: 0.26% to 1.05 %
- Strain rates: 4.5 x 10^{-3} to 6.2x10^{-6} s^{-1}
- Temperatures: 160 to 450 °C
- Oxygen concentrations: 1.0x10^{-5} to 6.0x10^{-10} wt.%
- Reference tests conducted under a high vacuum condition





d=3.2 mm
L_0=7 mm

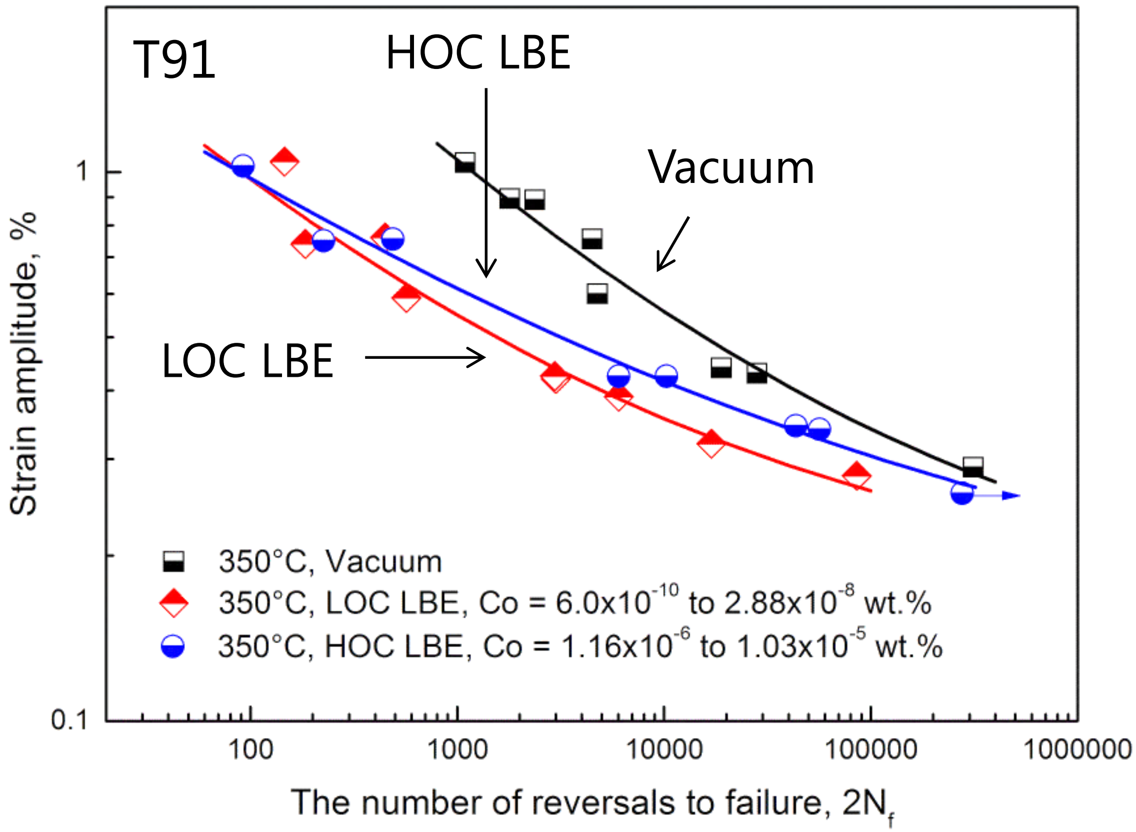
N_f: Number of cycles to failure
N_a: Number of cycles to initiate a macroscopic crack
N_f – N_a: indirectly represents fatigue crack propagation rate

LIMETS 3 fatigue testing system

Pierre Marmy, Xing Gong, Journal of Nuclear Materials, 2014, 450(1-3): 256-261

Effect of LBE environment on fatigue endurance

$\varepsilon - N$ diagram



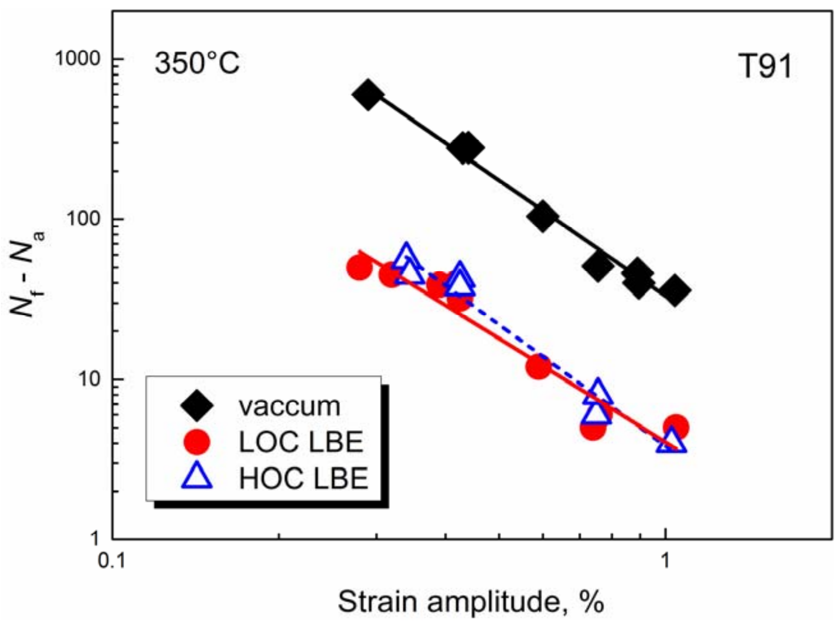
LOC: low oxygen concentration

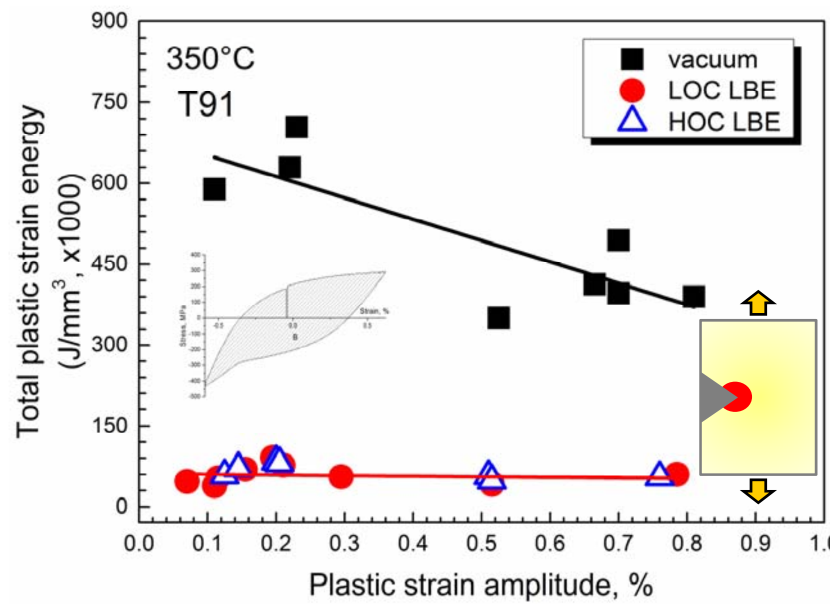
HOC: high oxygen concentration

- Fatigue endurance is strongly degraded in LBE, particulary at high strain amplitudes
- Oxygen concentration plays a role at low strain amplitudes

Effect of LBE environment on N_f - N_a

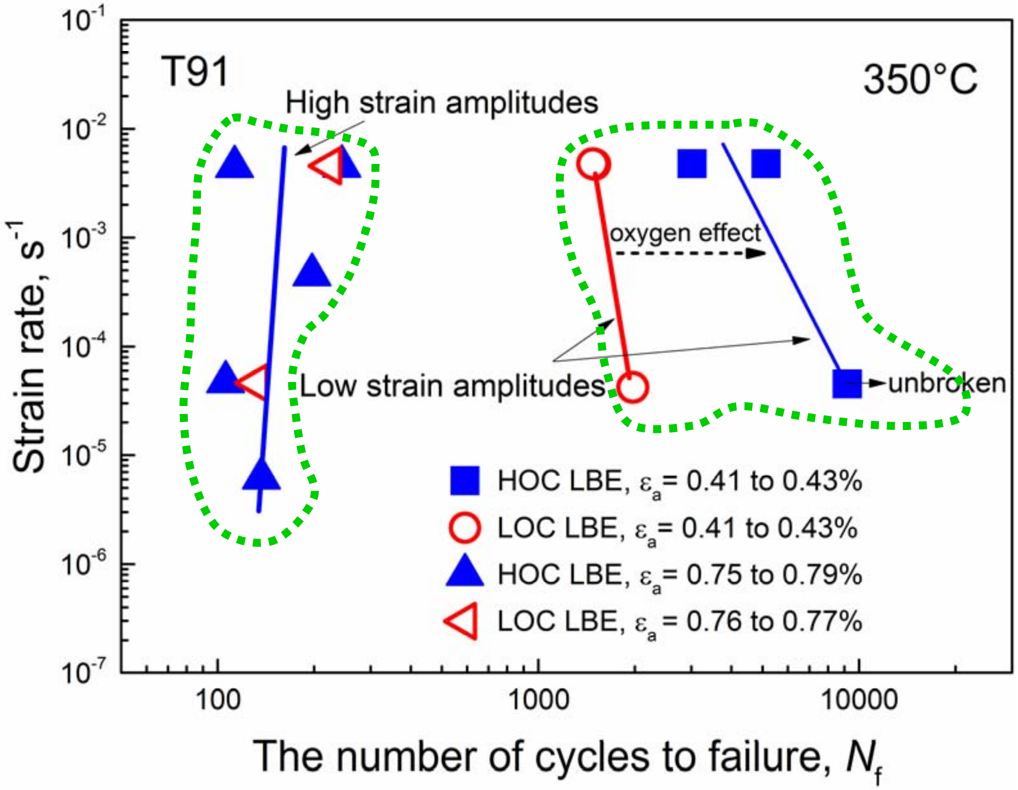
The area of a hysteresis loop is ***plastic strain energy density*** (kN/mm^2 or J/mm^3). The total plastic strain energy is the sum of the area of each hysteresis loop from N_a to N_f





The fatigue cracking in the presence of LBE is mainly brittle and is not strongly affected by oxygen concentration

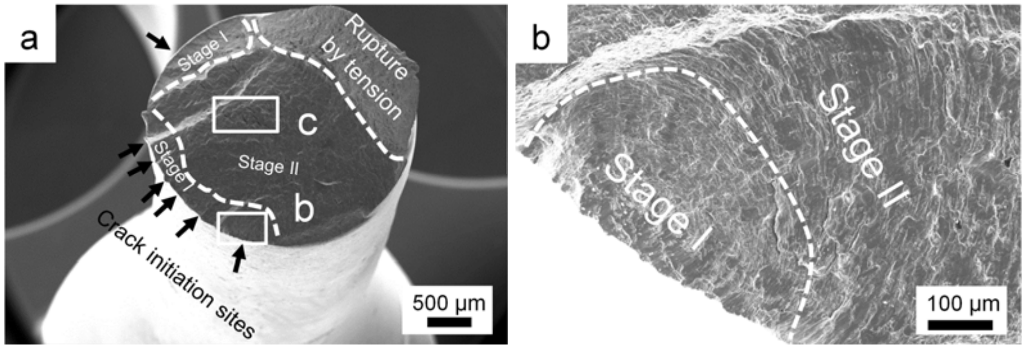
Effects of strain rate and oxygen concentration



- Strain rate effect seems to be strain amplitude dependent
- At high strain amplitudes, fatigue life is not significantly influenced by oxygen concentration, but a pronounced oxygen effect is present at low strain amplitudes
- Significant fatigue life improvement is observed under a combination of low strain amplitude, slow strain rate and high oxygen

Fractography

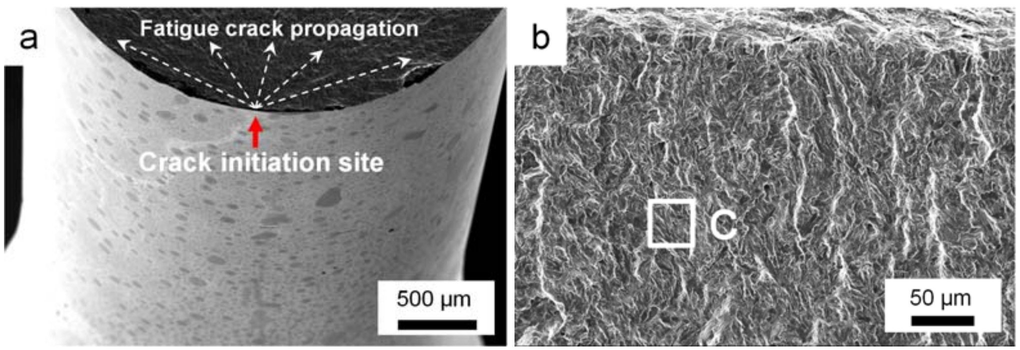
350°C, vacuum,
ε_a = 1.04%



In vacuum:

- Rough fracture surface
- Multiple crack initiation sites
- A clear transition from Stage I and Stage II

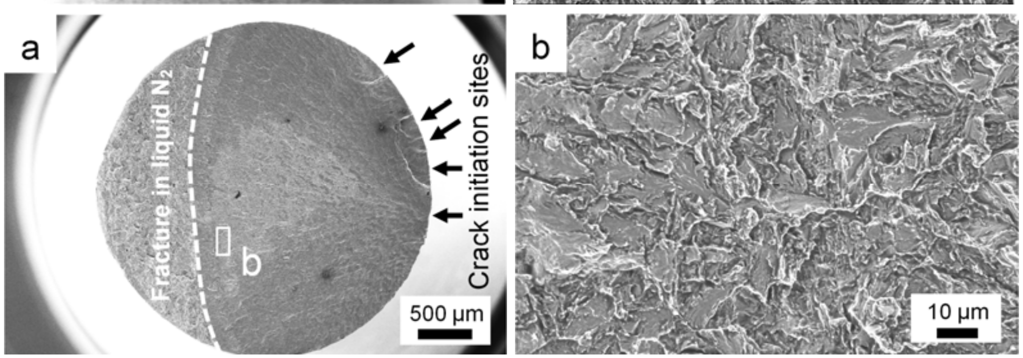
350°C, LOC LBE,
Co = 2.88x10^{-8} wt.%, ε_a = 0.59%



In LOC LBE:

- Flat fracture surface
- Unique crack initiation site
- **No Stage I**
- **Quasi-cleavage**

350°C, HOC LBE,
Co = 4.12x10^{-6} wt.%, ε_a = 1.03%

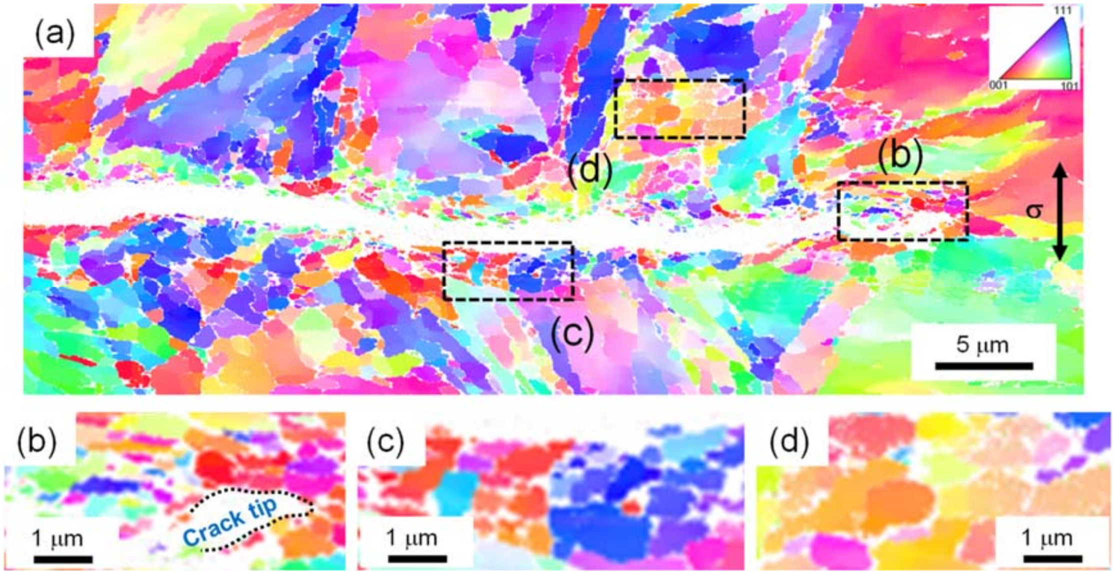


In HOC LBE:

- Flat fracture surface
- **Multiple crack initiation sites**
- **No Stage I**
- **Quasi-cleavage**

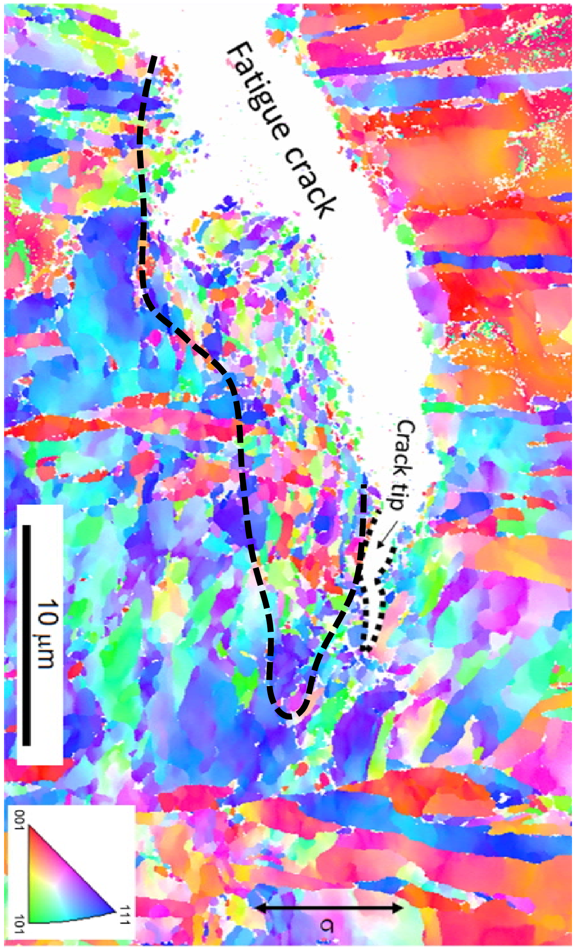
Cyclic plasticity induced grain refinement in vacuum

T91, 350°C, vacuum, ε_a = 0.43%



ND inverse pole figure coding

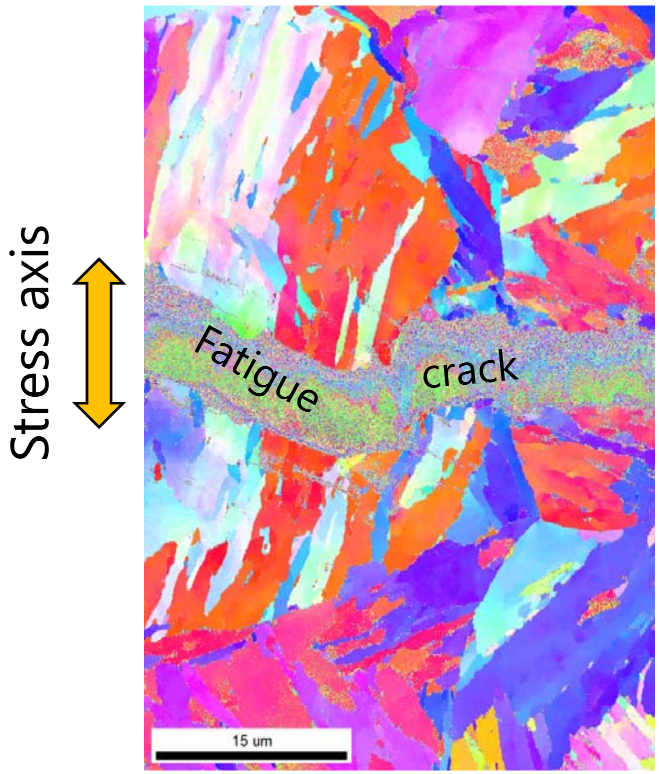
T91, 350°C, vacuum, ε_a = 0.9%



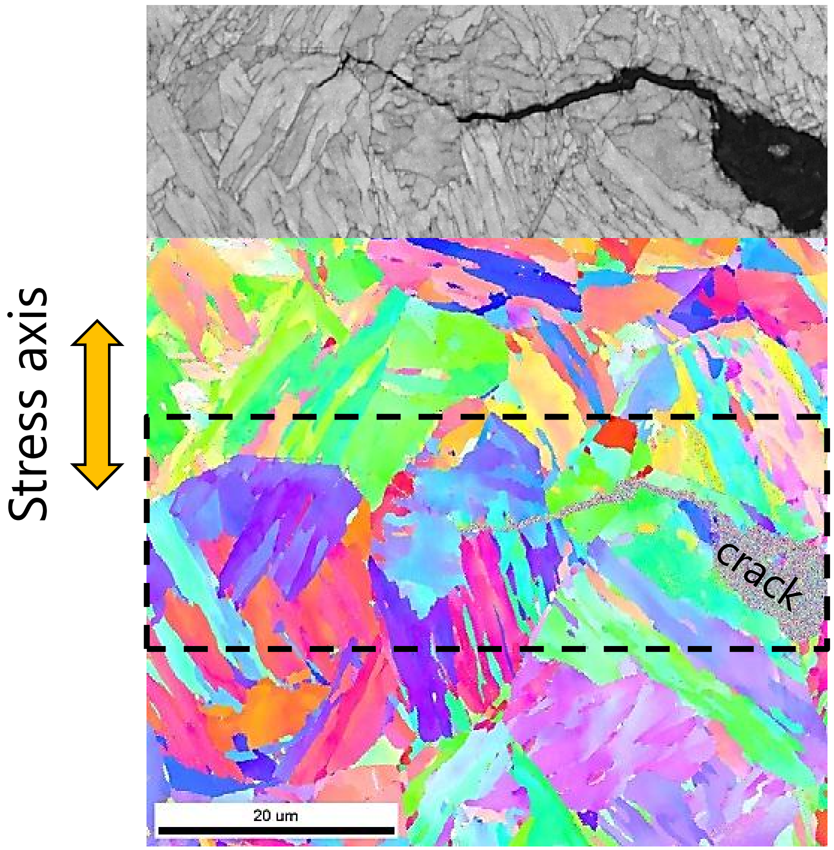
- Numerous equiaxed subgrains evolved from pre-existing martensite – cyclic plasticity induced grain refinement
- Indicating **highly localized plastic shear strain** around the crack tip

Absence of strong plastic shear strain localization in LBE

350°C, LBE, C_O = 2.2x10^{-8} wt.%,
ε_a = 0.42%



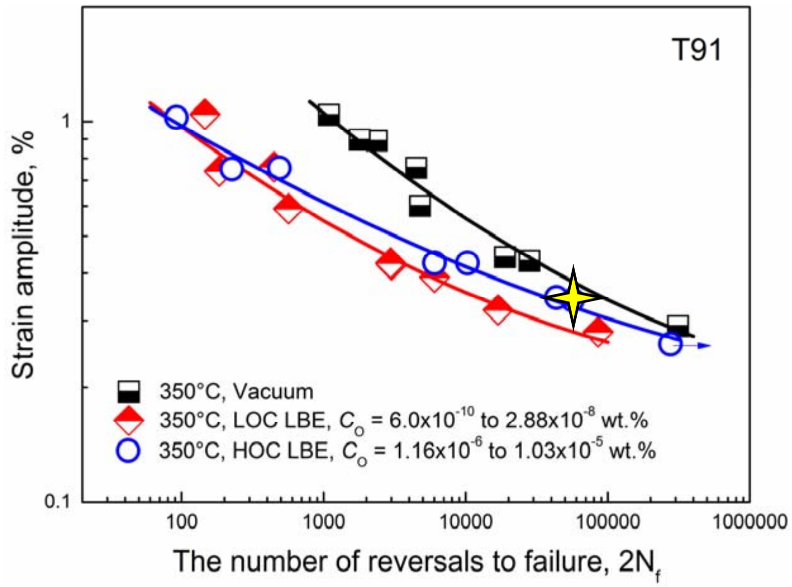
350°C, LBE, C_O = 1.16x10^{-6} wt.%,
ε_a = 0.32%

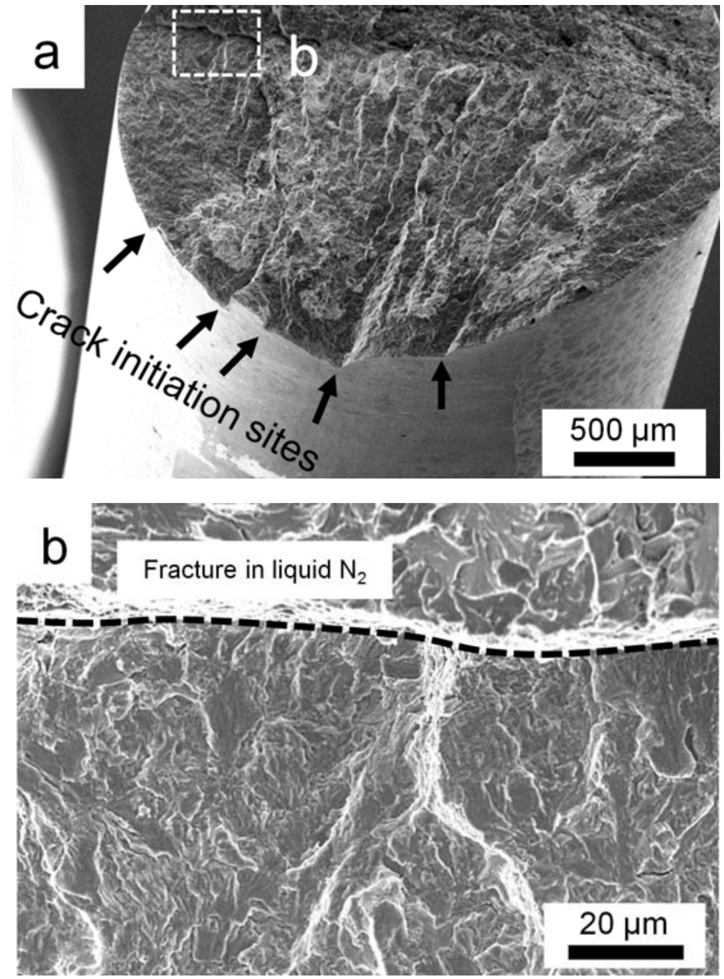


No evident grain refinement → Limited plastic deformation around the crack tip in the presence of LBE

Disappeared LME or …?

350°C, HOC LBE, C_O= 5.85x10^{-6} wt.%, ε_a = 0.35%



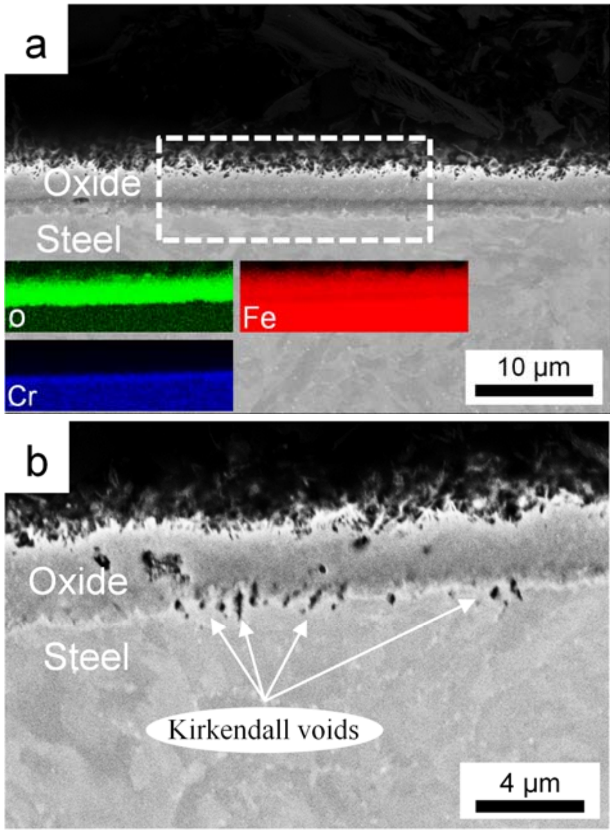


LME does not disappear, evidenced by the observation of quasi-cleavage

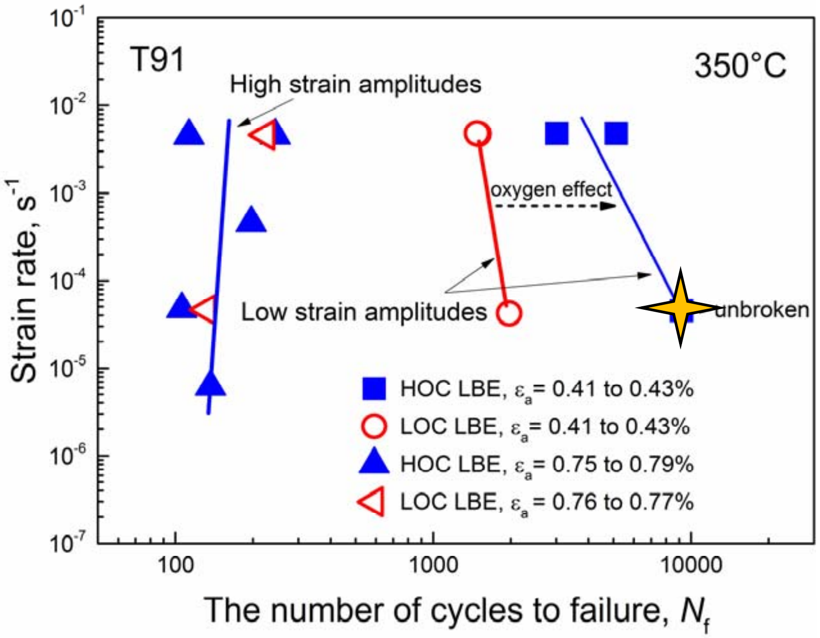
However, fatigue crack initiation mechanism may be modified due to high oxygen concentration

Surface protection by oxide films

350°C, HOC LBE, C_O = 2.6x10^{-5} wt.%, ε_a = 0.41%, strain rate=4.5x10^{-5} s^{-1}



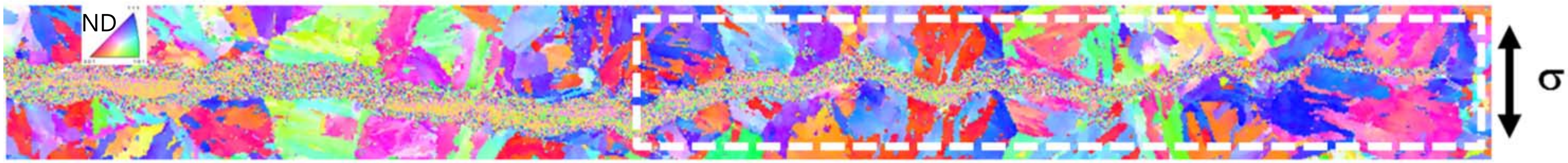
Double-layer oxides



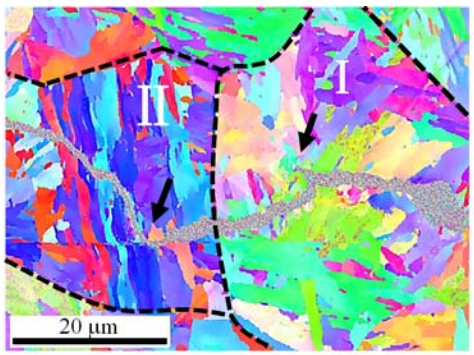
Oxide protection delays fatigue crack initiation under a combination of small strain amplitude, slow strain rate and high oxygen condition

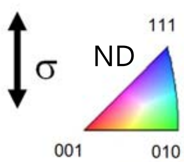
Fatigue crack propagation modes

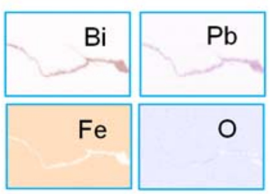
350°C, LBE, C_O = 2.2x10^{-8} wt.%, ε_a = 0.42%

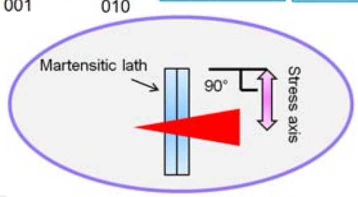


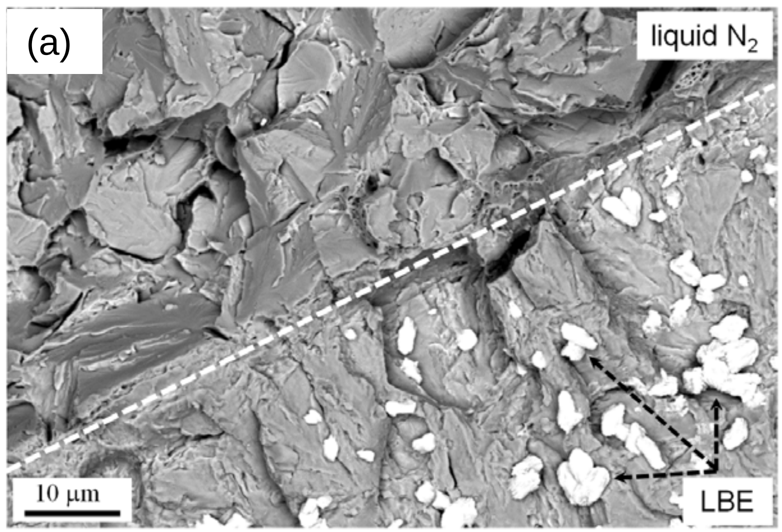
350°C, LBE, C_O = 1.16x10^{-6} wt.%, ε_a = 0.32%



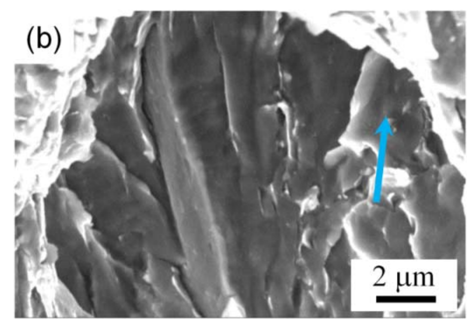






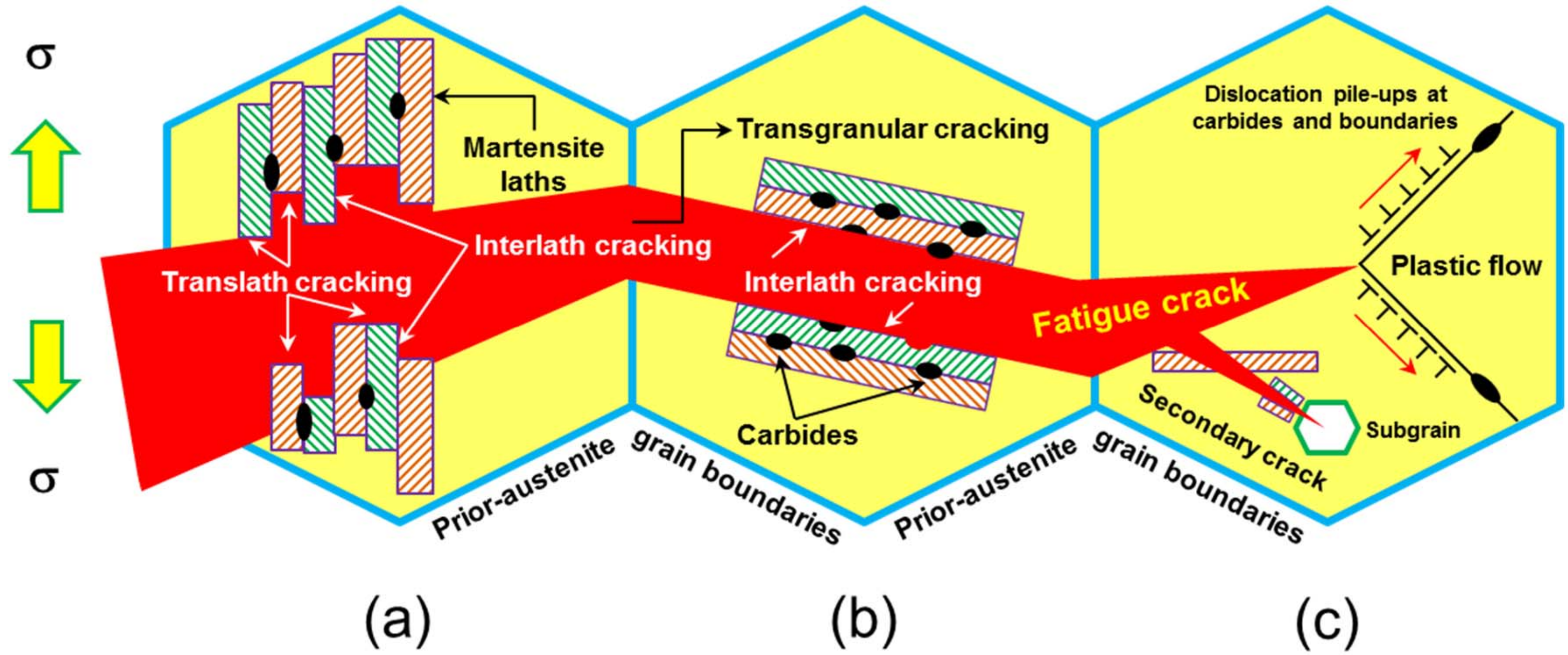


350°C, LBE, C_O = 2.24x10^{-8} wt.%, ε_a = 1.05%



- The main crack propagates through the prior-austenite grains - **transgranular**
- **Intergranular** or **interlath cracking** occurs when the boundaries are oriented stress-favourably → **Orientation dependent**

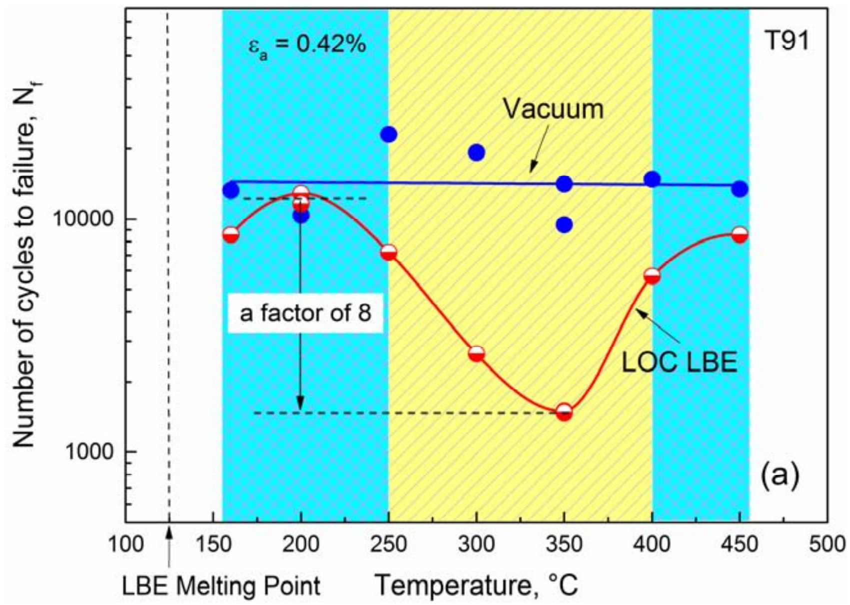
Fatigue crack propagation modes - Summary

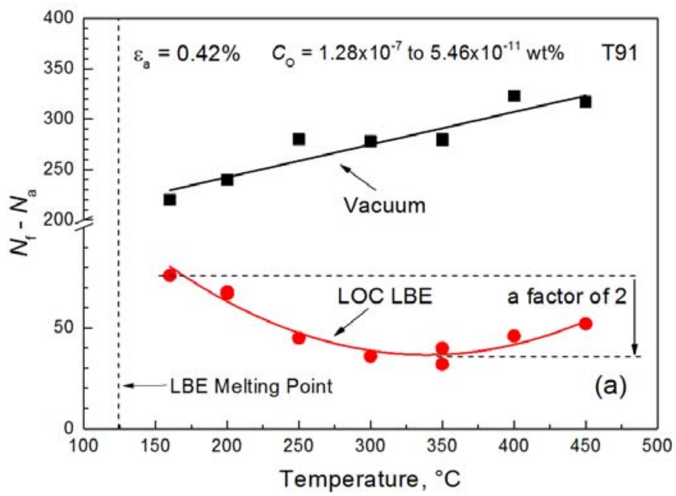


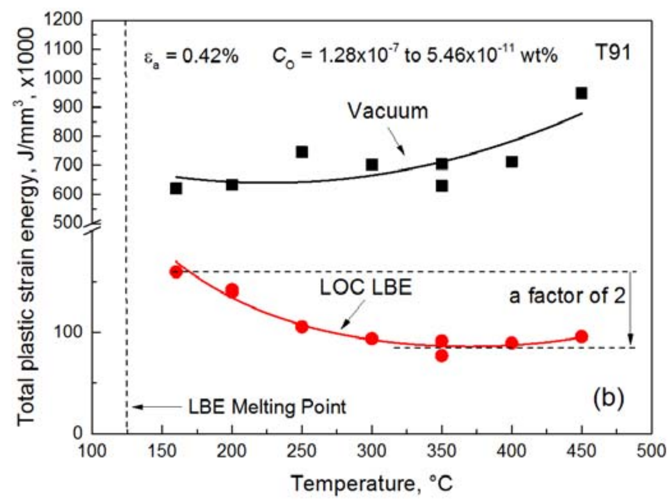
(a) Alternate occurrence of interlath and translath cracking, oriented stress-unfavourably
(b) Pure interlath cracking, oriented stress favourably
(c) Secondary cracking and limited plastic flow in the vicinity of the crack tip

Indication of "Critical stress criterion"

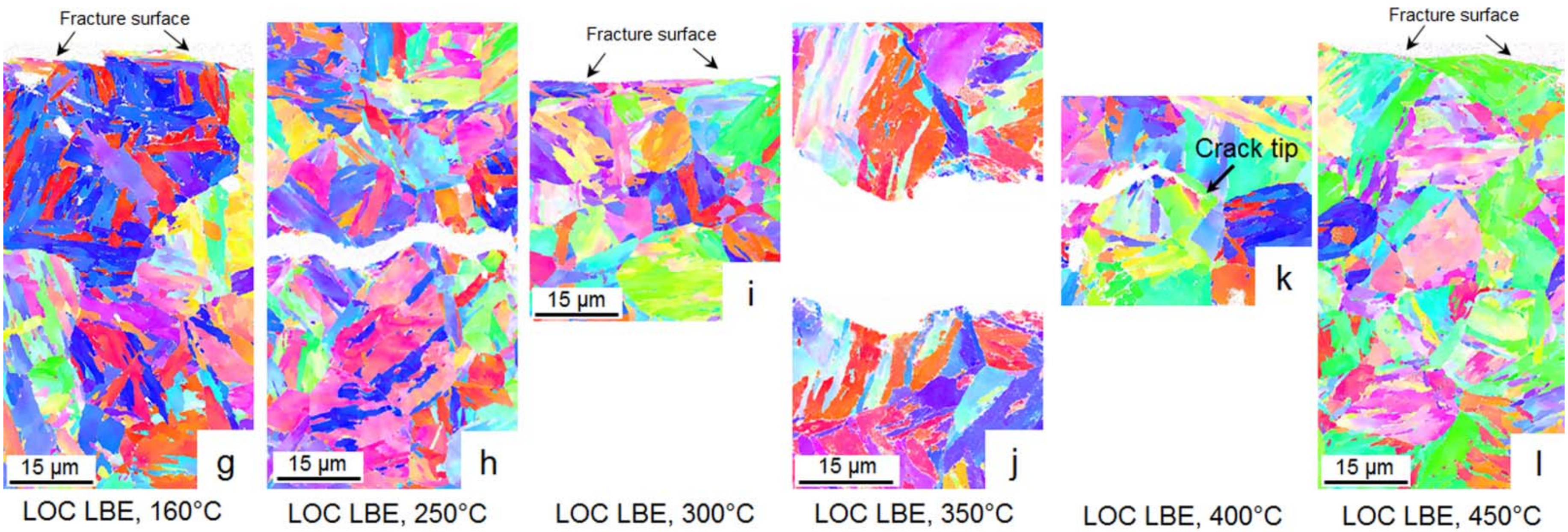
Fatigue endurance “trough”







Microstructure close to fracture surface or fatigue crack walls



In vacuum

Obvious grain refinement

→ strong plastic shear strain localization around the crack tip

In LOC LBE

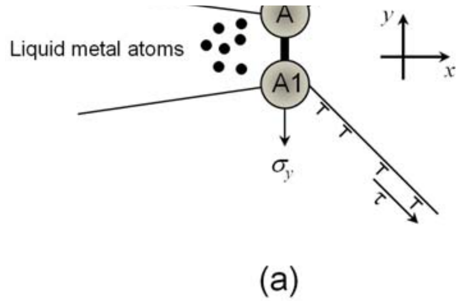
No evident grain refinement

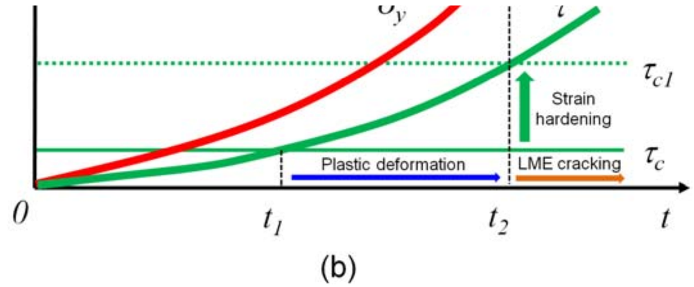
→ **limited plastic deformation around the crack tip due to LME**

⇨ LME can occur at all the temperatures investigated during the fatigue crack propagation!!

A proposed new LME mechanism (1/2)

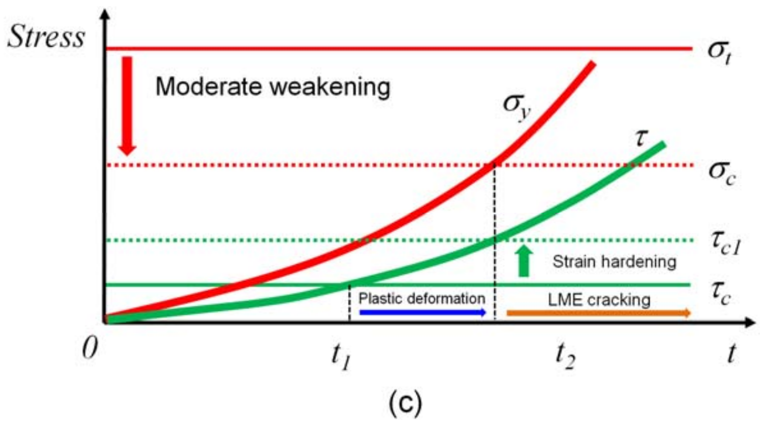
- Orientation dependence of boundary cracking – Critical stress criterion
- Quasi-cleavage – a combination of brittle and ductile features

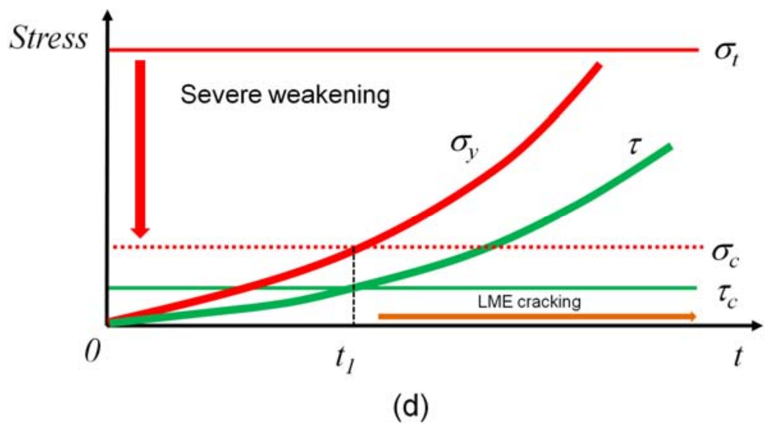




(a) Adsorption of liquid metal atoms (Stoloff_1963 and Westwood_1963)

(b) **Slight weakening** – strong strain hardening is required for LME





(c) **Moderate weakening** – slight strain hardening is required for LME

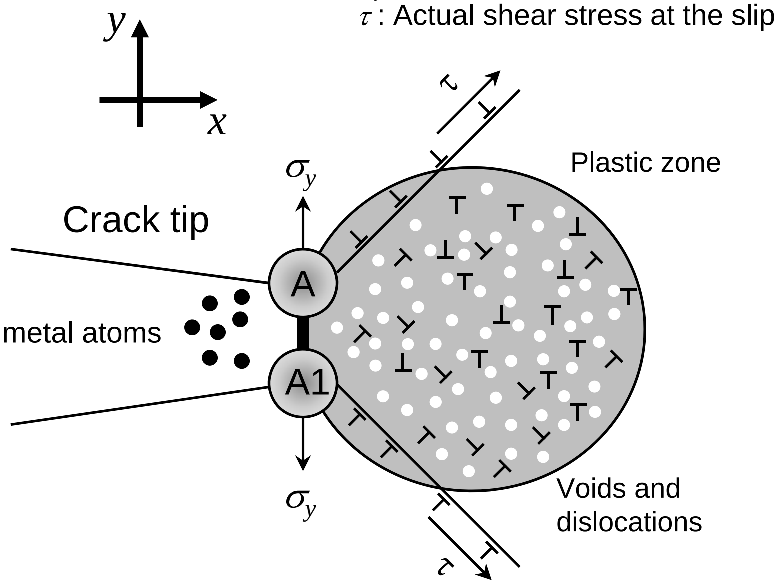
(d) **Severe weakening** – plastic deformation and strain hardening are not required for LME

A proposed new LME mechanism (2/2)

Mechanism of the formation of quasi-cleavage

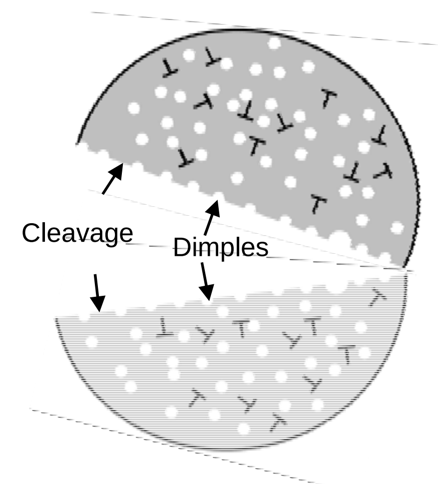
σ_c: Critical normal stress to break the atomic bond
τ_c : Critical resolved shear stress to activate dislocation glide at a given slip plane
σ_y : Actual normal stress acting on the atomic bond A-A1
τ : Actual shear stress at the slip plane

(a)



- **Moderate weakening** by liquid metal atoms → σ_c is reduced, τ_c is not influenced, but σ_c remains higher than τ_c
- τ first reaches τ_c but σ_y is still lower than σ_c → Plastic deformation occurs and plastic strain is localized into the plastic zone → Dislocations, voids and other defects are formed in the plastic zone

(b)



- Accumulation of the defects in the plastic zone → **Strain hardening** → τ_c climbs up
- τ gets difficult to overcome the increased τ_c, but σ_y starts to reach σ_c → The A-A1 atomic bond is broken and rupture occurs through the hardened plastic zone → A mixture of tiny dimples and cleavage features is formed → **Quasi-cleavage**
- The same process repeats until the final failure of the specimen

Conclusions

- Fatigue life of T91 steel is substantially reduced due to LME but the extent of the fatigue life reduction depends on oxygen concentration, strain amplitude and strain rate
- Oxygen concentration effect is strain amplitude dependent. A high oxygen condition can decrease the extent of the fatigue life reduction at low strain amplitudes
- Strain rate has no strong effect on the fatigue life at the high strain amplitudes, but the fatigue life is greatly improved under the condition of low strain amplitudes and a high oxygen condition and a slow strain rate
- The oxygen effect might be associated with surface oxide protection. Due to this effect, the fatigue crack initiation is delayed
- Intense plastic shear strain localization is observed around the crack tip in vacuum, manifested by the formation of very fine subgrains in the vicinity of the crack walls. By contrast, no obvious microstructures related to plastic shear strain localization can be observed around the crack tip in the presence of LBE
- Transgranular and translath cracking are the predominant failure modes for the LME-affected fatigue cracks. Interlath or intergranular cracking occationally occurs and their occurrence is orientation dependent relative to the stress axis
- A fatigue endurance “trough” is found to be present at 350°C
- The susceptibility of LME might be linked to the extent of atomic cohesion weakening. A new LME mechanism is proposed to explain the formation of quasi-cleavage

Thanks for your attention

Questions?