

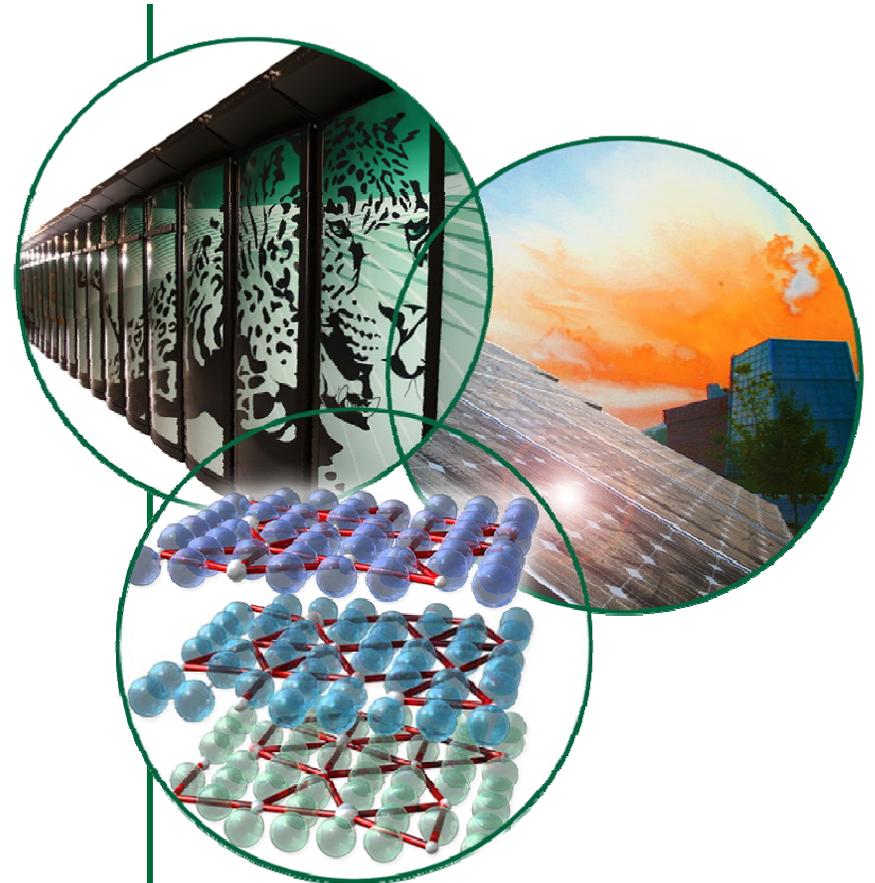
# Creep-Fatigue-Oxidation Interactions: Predicting Alloy Lifetimes under Fossil Energy Service Conditions

***2016 NETL Crosscutting  
Research Review Meeting  
April 18-22 2016, Pittsburgh***

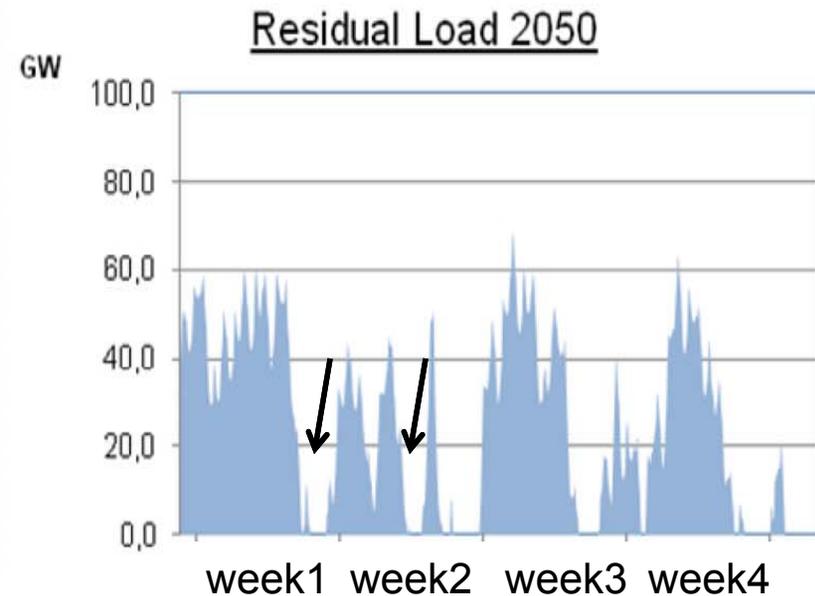
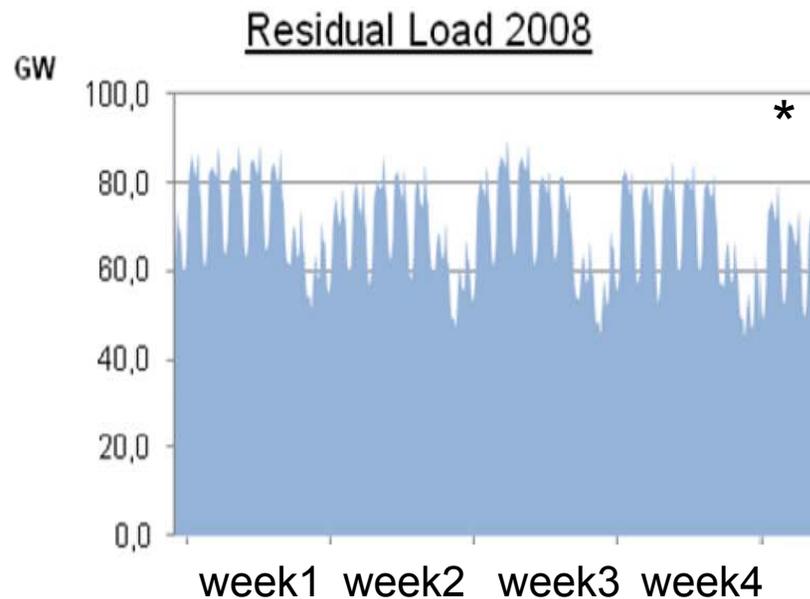
**Sebastien Dryepondt**

**Amit Shyam**

***Oak Ridge National Laboratory***



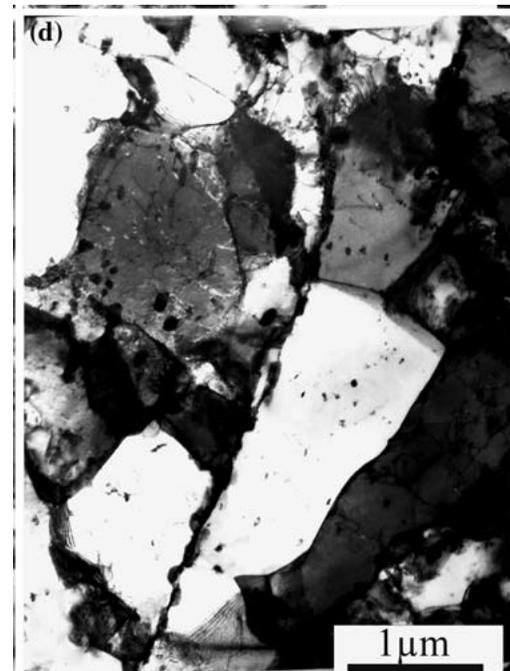
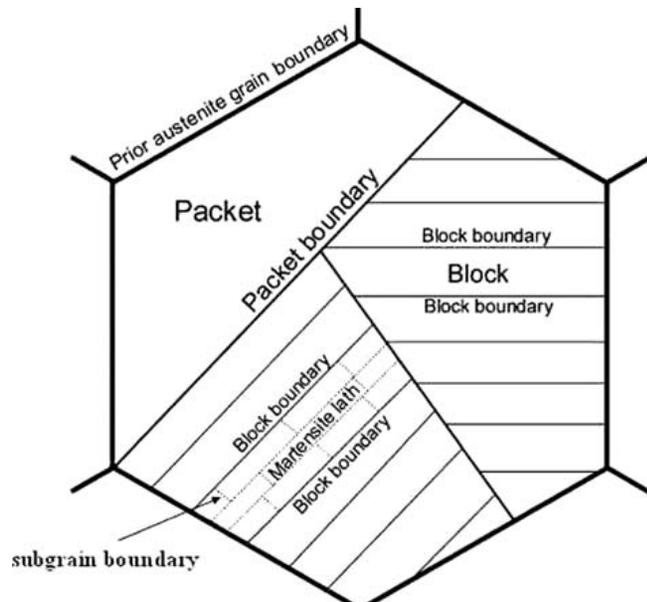
# Power Plants Will Need to be Capable of Flexible Operation



- Frequent (~daily) load cycling will result in significant creep-fatigue interaction
- Project will focus on:
  - Long term creep fatigue testing and lifetime modeling
  - Study of microstructurally small cracks under creep-fatigue loading
  - Interactions among creep fatigue and oxidation

# Creep-Fatigue Behavior of Gr.91 Steel

- Gr.91 creep performance is significantly affected by cyclic loading due to microstructure changes (Fournier et al. 2008)

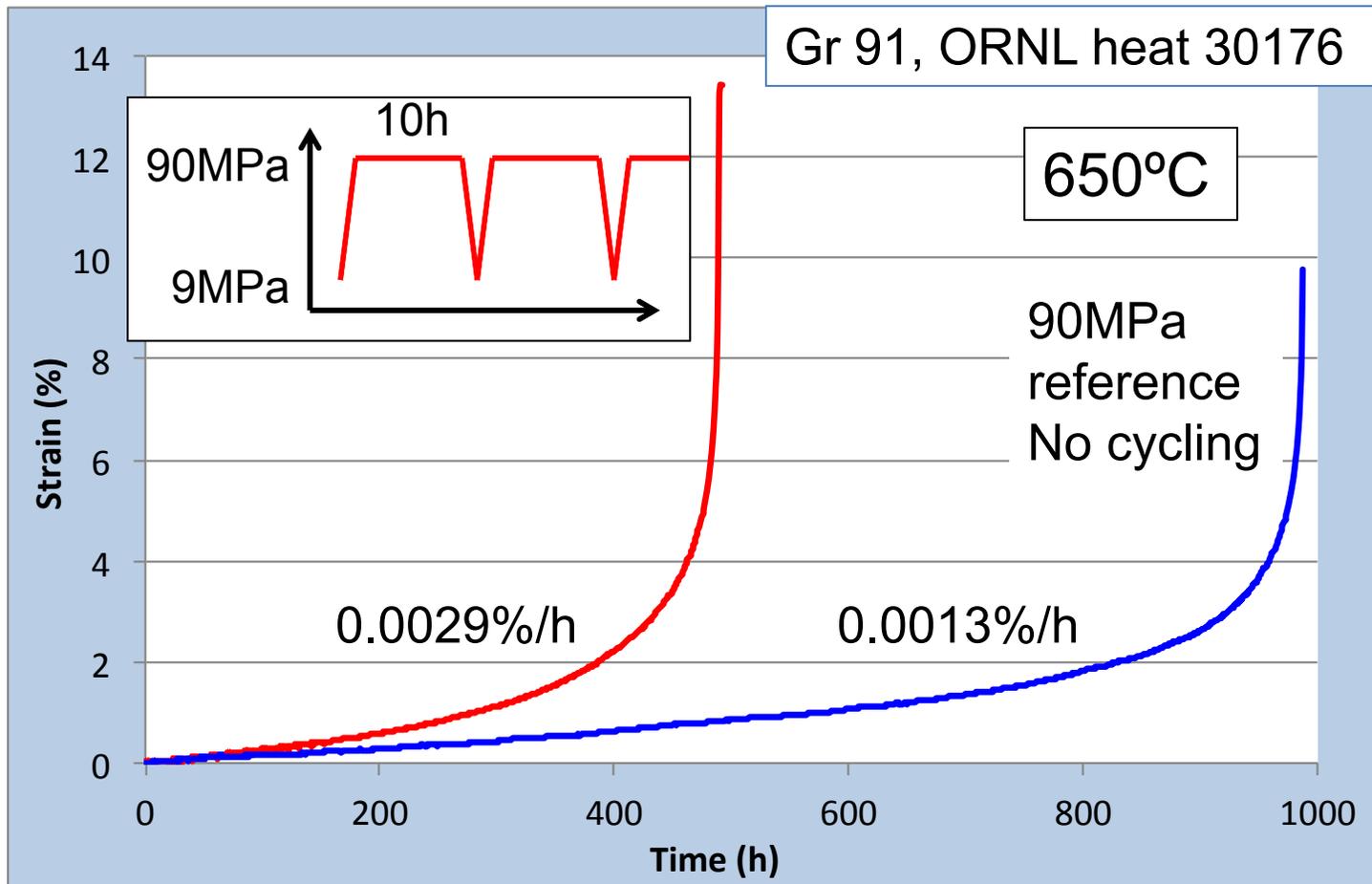


As Received      Creep-Fatigue Tests at 550°C

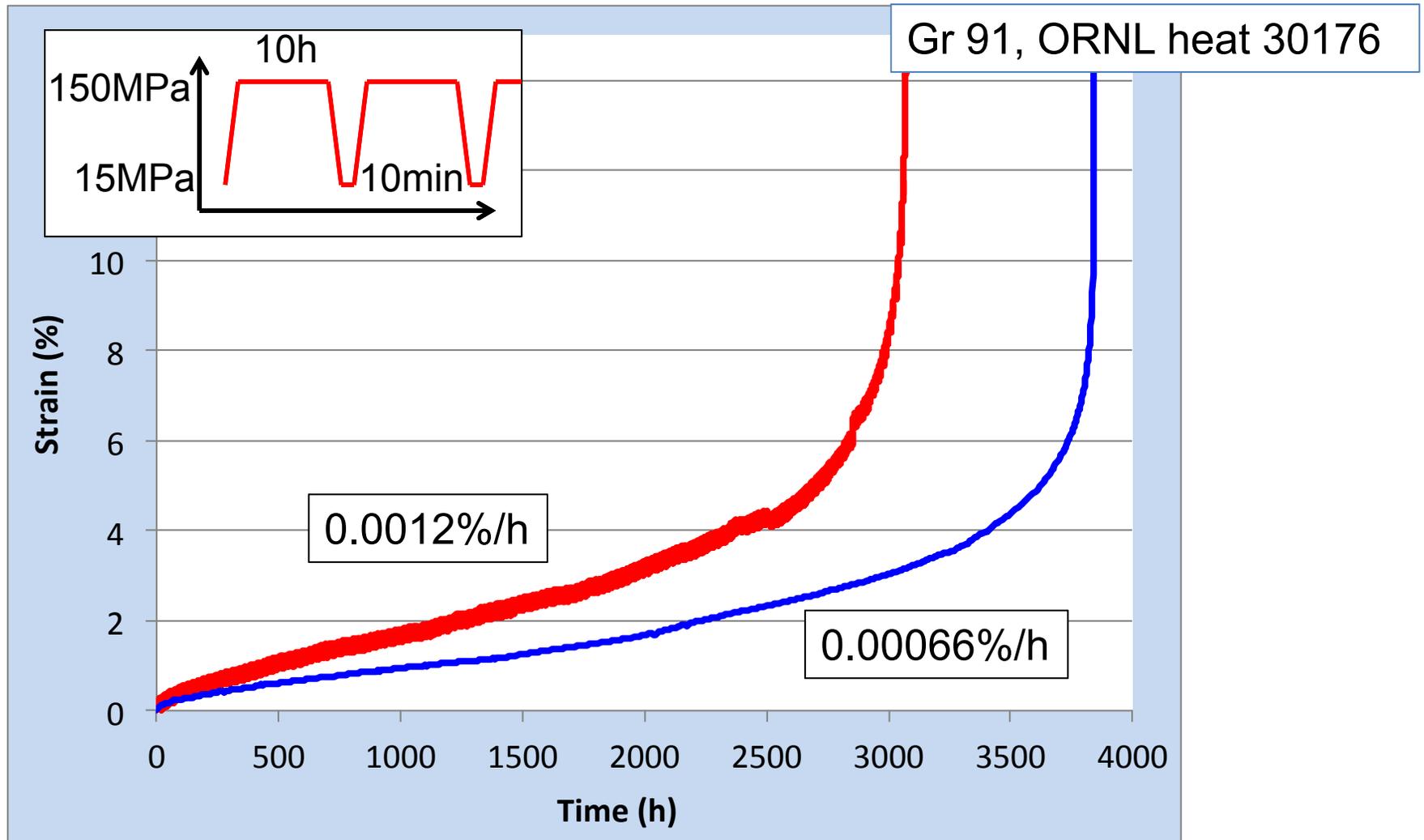
- sub grain coarsening
- dislocation density decrease

- No significant effect of cyclic creep (unloading/loading) due to limited cyclic strain ( 3500h, ~50 cycles)

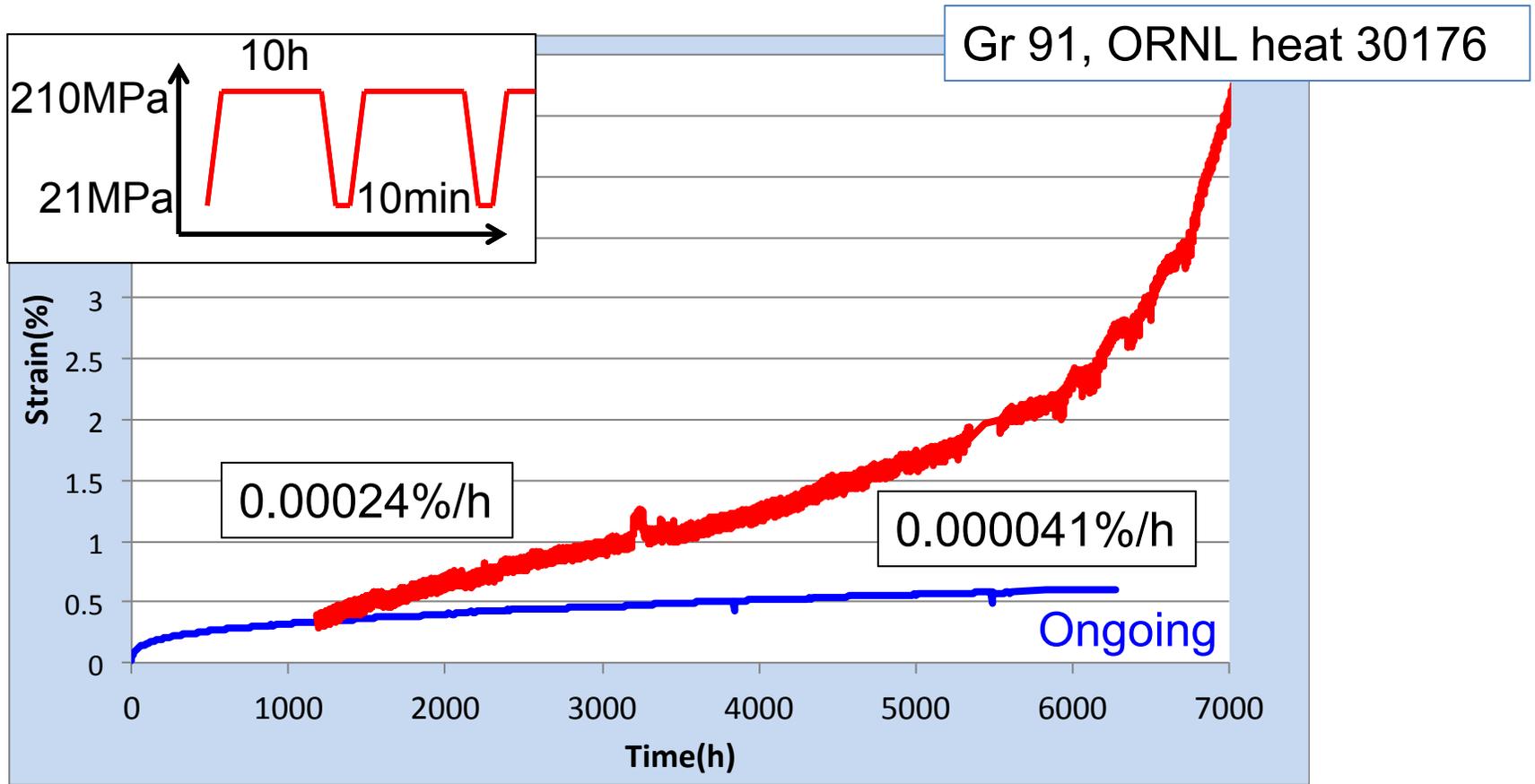
# Decrease of lifetime at 650°C, 90MPa with 10h cycle



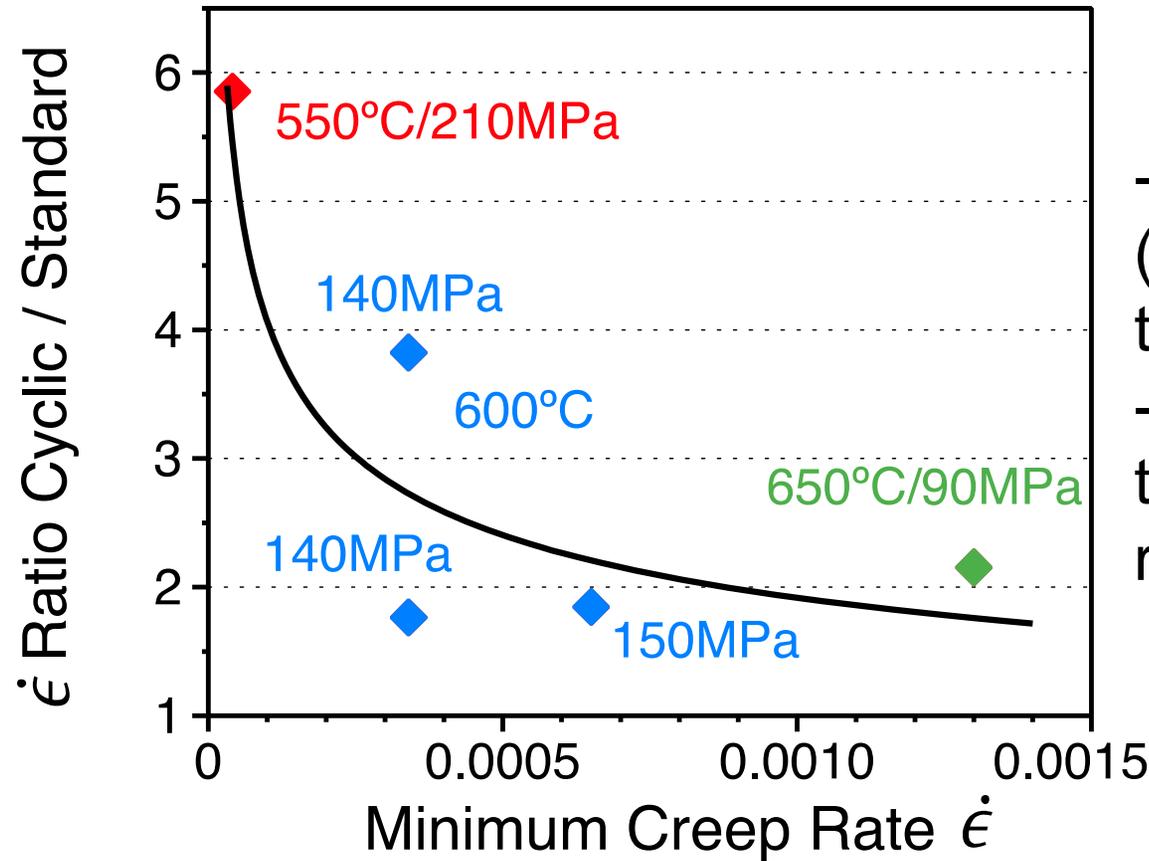
# Increase of creep rate at 600°C, 150MPa with 10h cycle



# Significant increase of creep rate at 550°C, 210MPa with 10h cycle

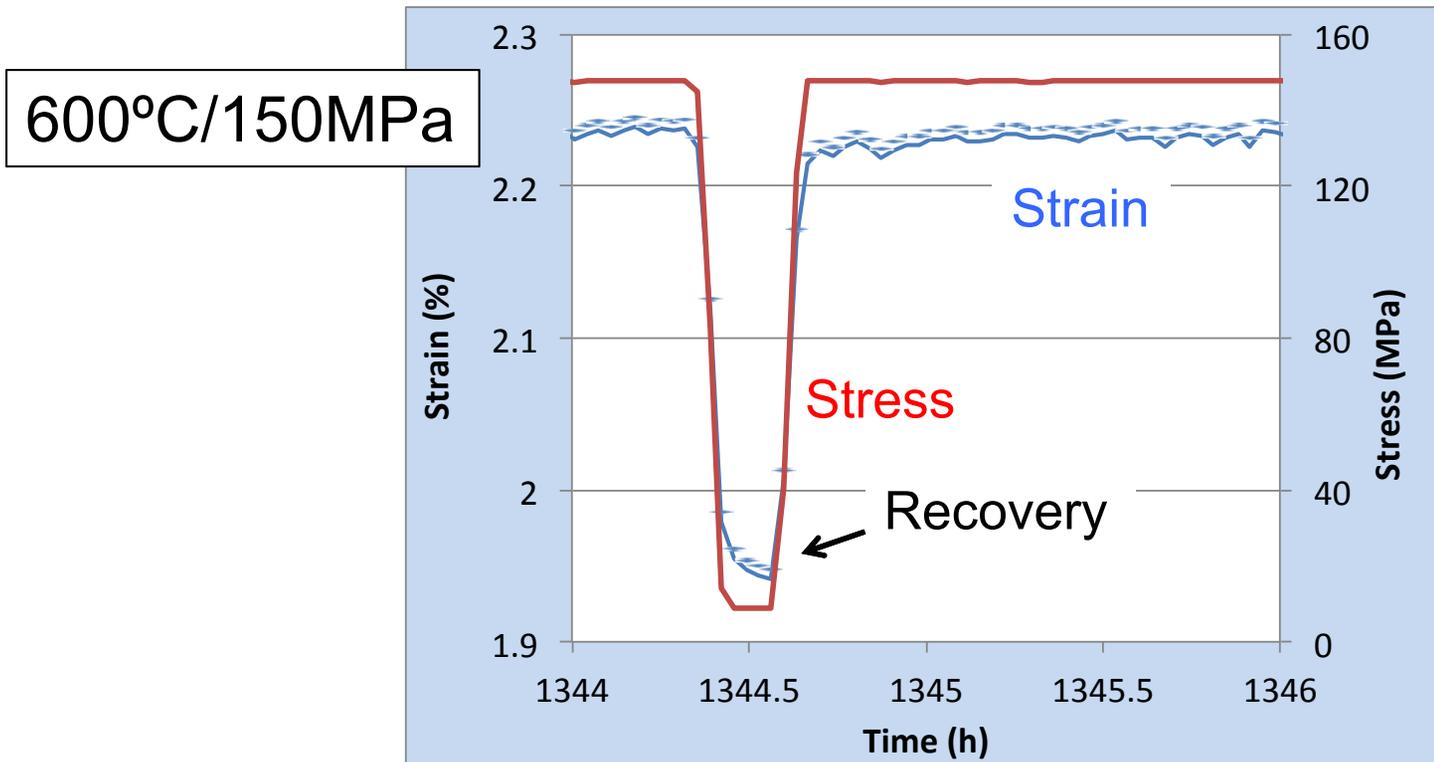


# Significant Increase of Creep Rate for Longer Creep Tests



- Systematic increase (~X2) of creep rate due to cycling
- Need to focus on long term test / low creep rate

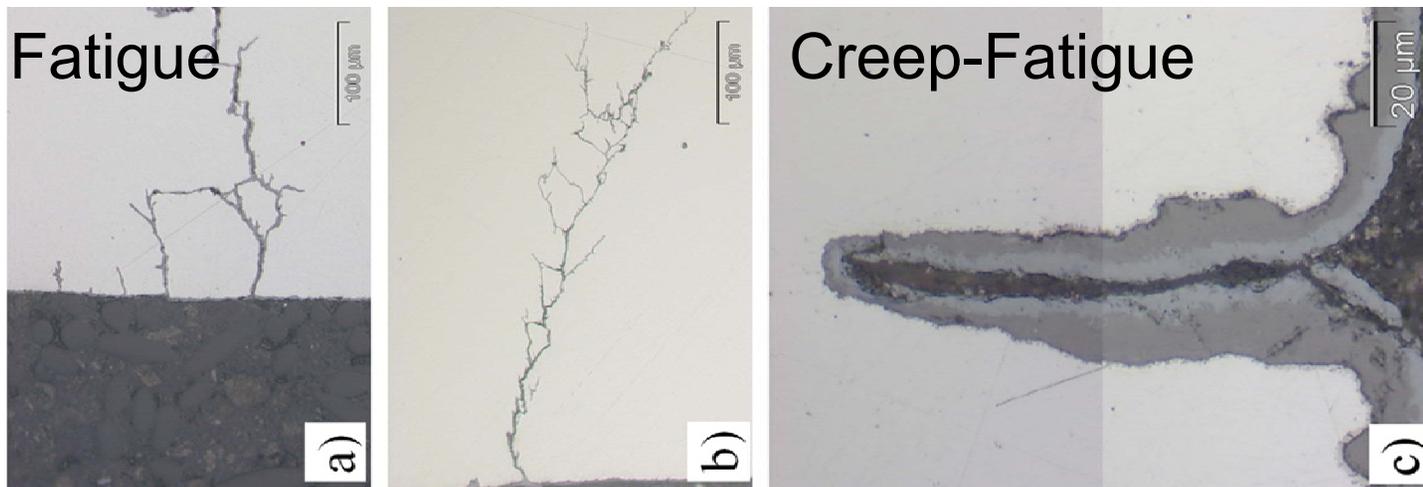
# Recovery Observed During Unloading at Each Cycle



Decrease of dislocation density?  
Coarsening of sub-grain structure?

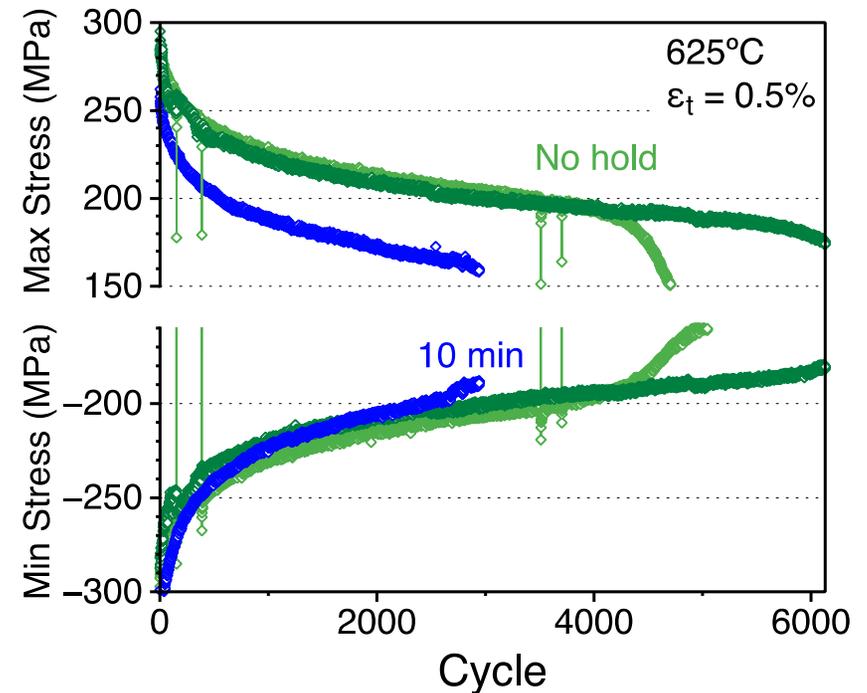
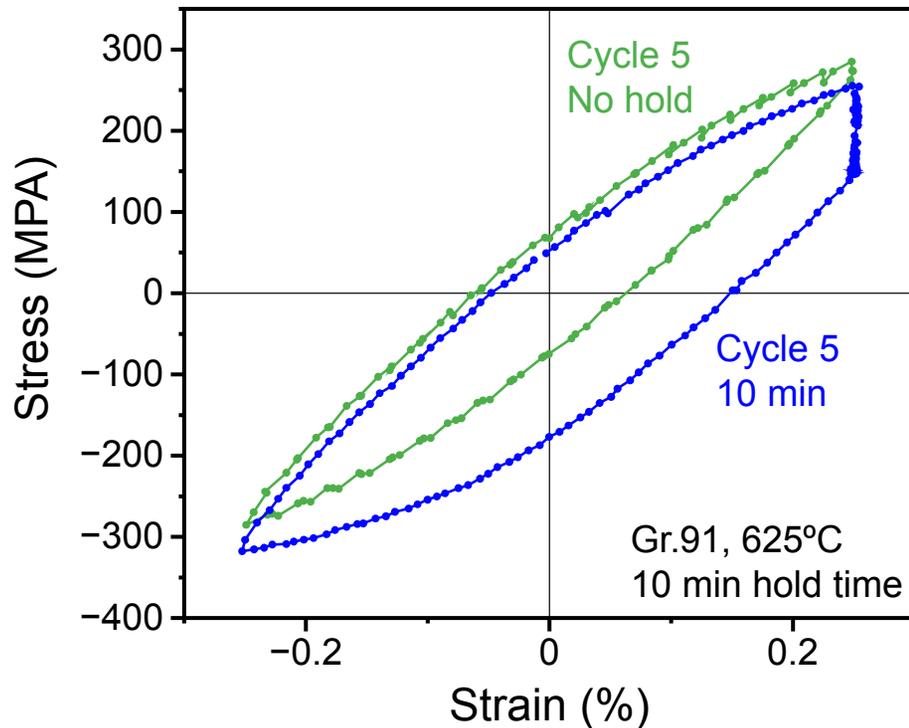
# Creep-Fatigue Behavior of Gr.91 Steel

- Decrease of cycle to rupture with hold time
- No cavity observed after testing
- Main effect of hold time is related to the formation of multi-layer thick scale vs thin scale on pure fatigue specimens
- **Need for longer creep fatigue test duration**



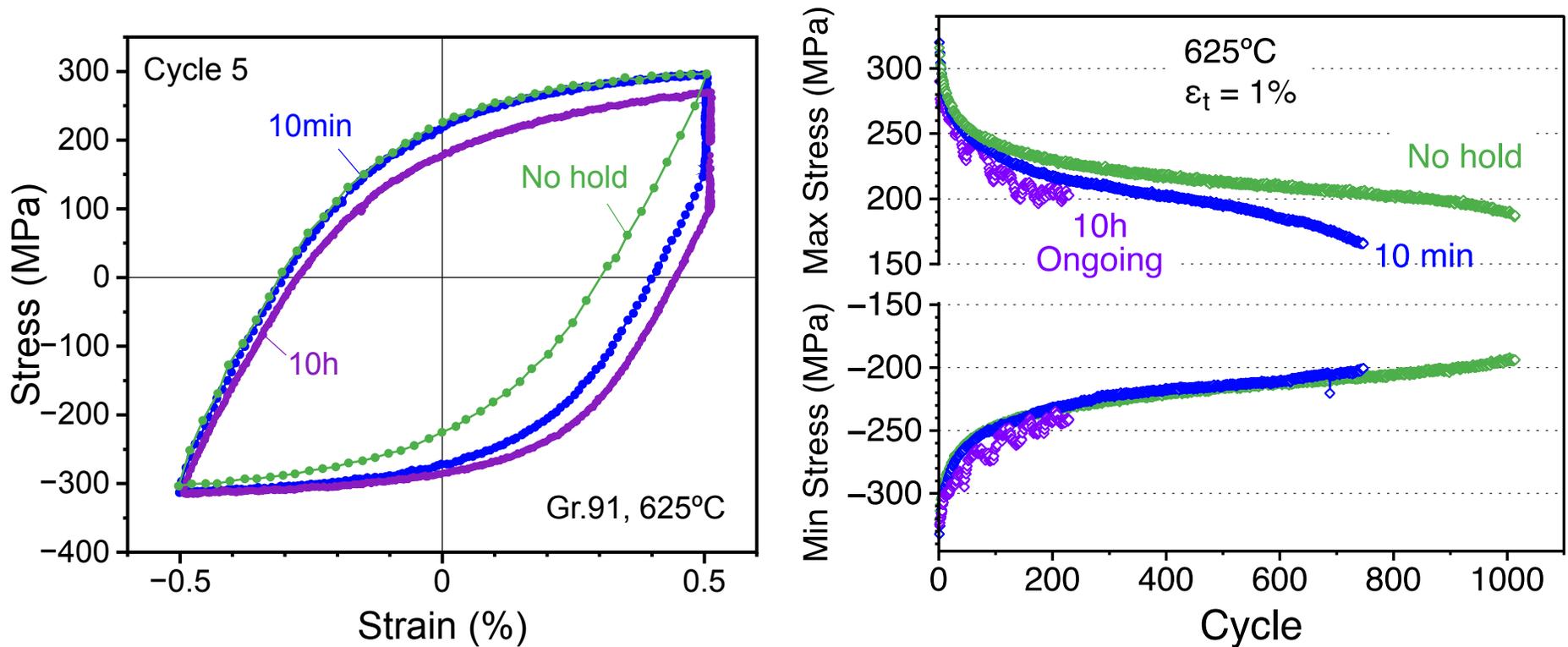
Fournier et al. (2008)

# Decrease of $N_f$ with Hold Time for Creep-fatigue Tests at 625°C, $\pm 0.25\%$



Faster softening due to 10min hold time (500h test)

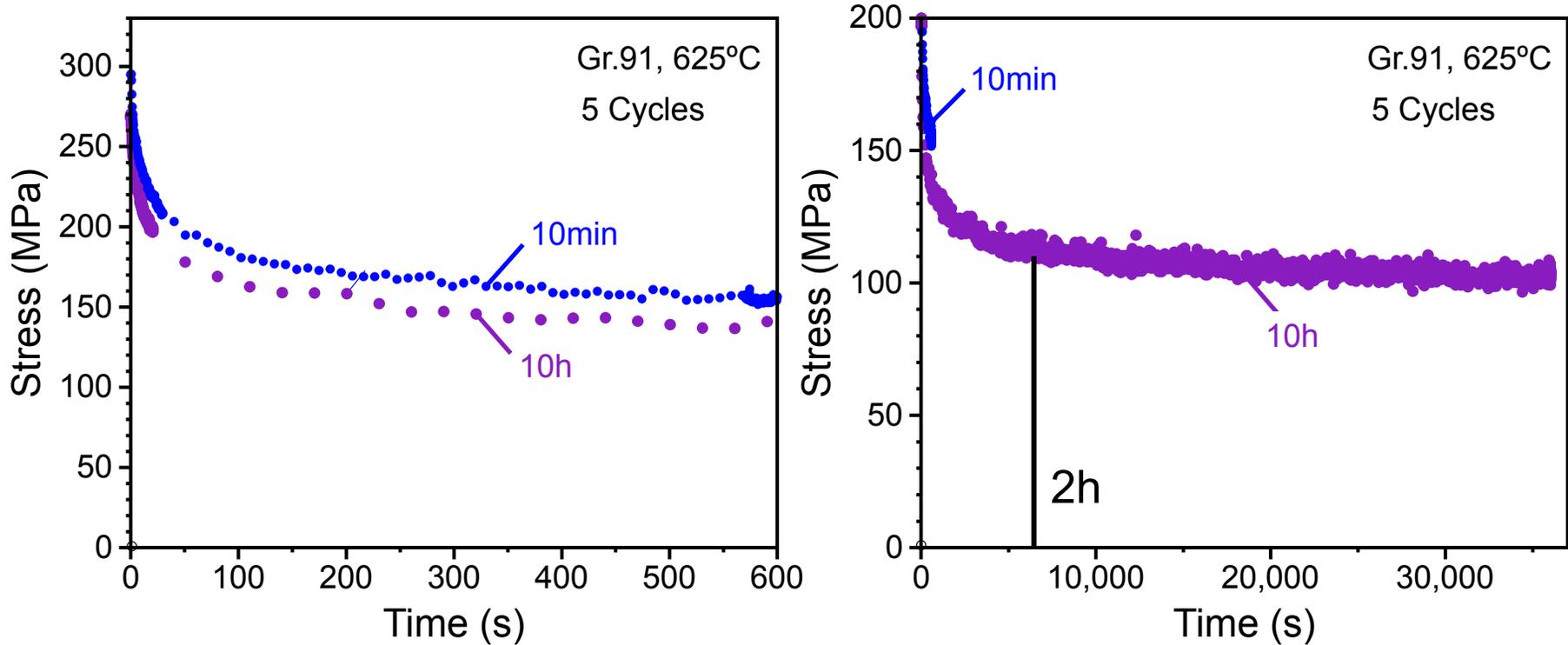
# Decrease of Nf with Hold Time for Creep-fatigue Tests at 625°C, ±0.5%?



-Faster softening due to hold time but not as drastic as for  $\pm 0.25\%$

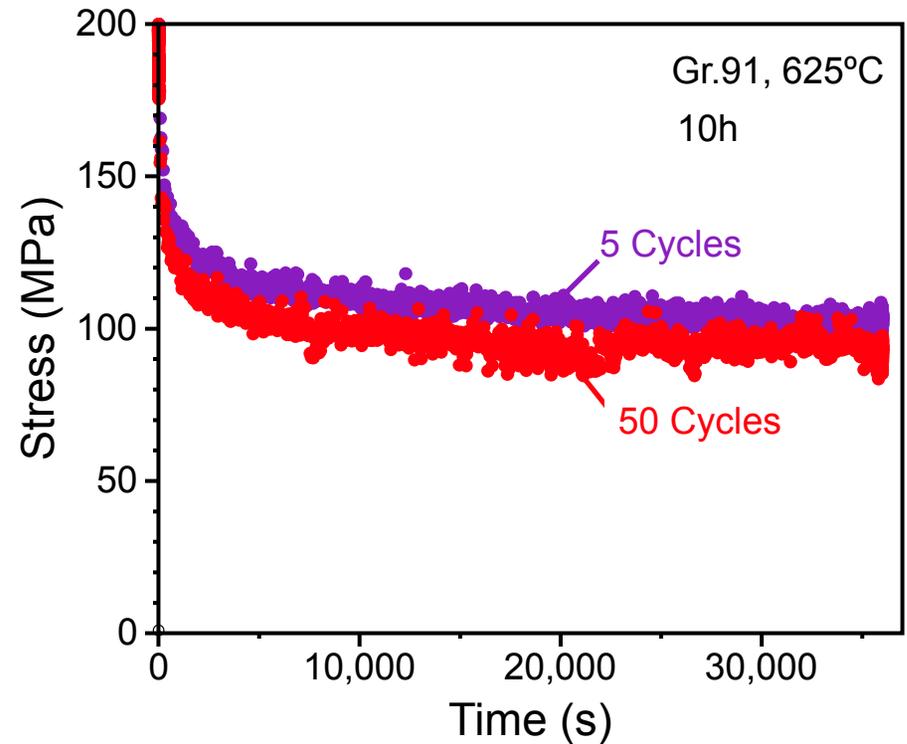
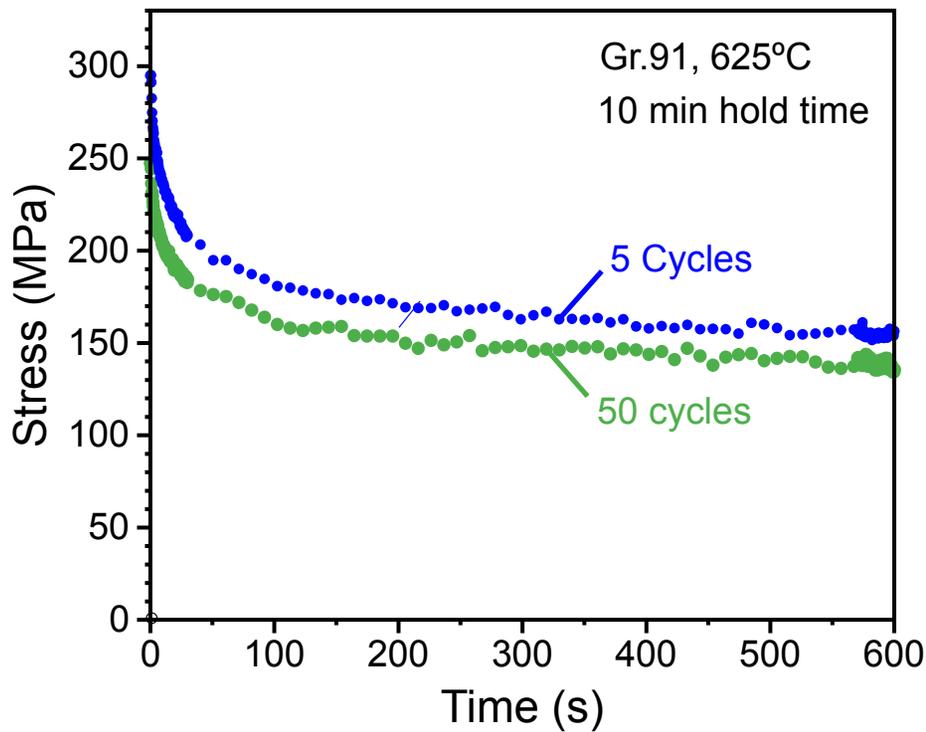
-Increase of noise for 10h hold test (>2100h)

# Significant Decrease of Stress During 10h Hold Time

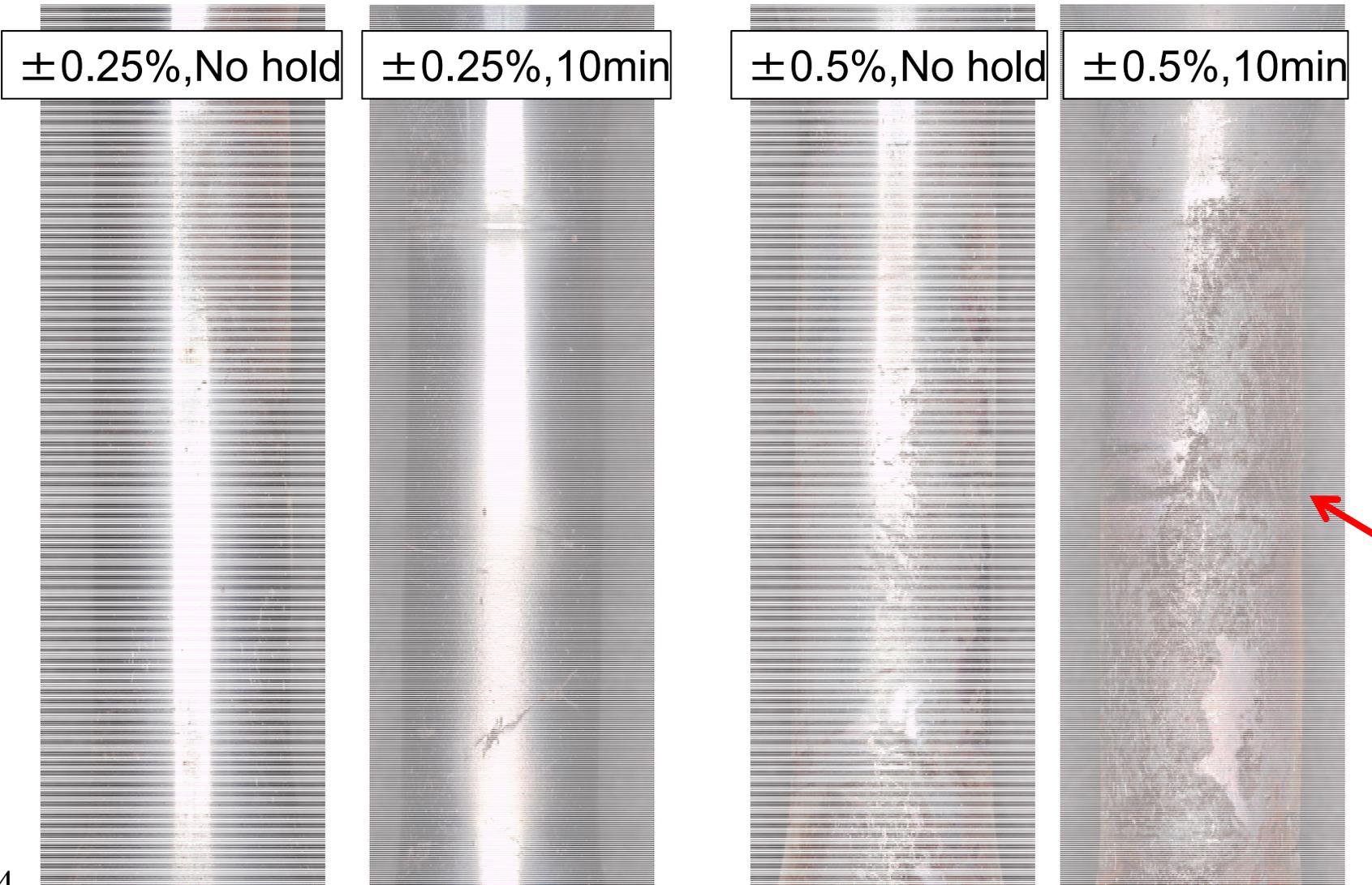


- Close to a steady stress after ~2h
- Creep lifetime at 625°C, 100MPa ~ 5000h

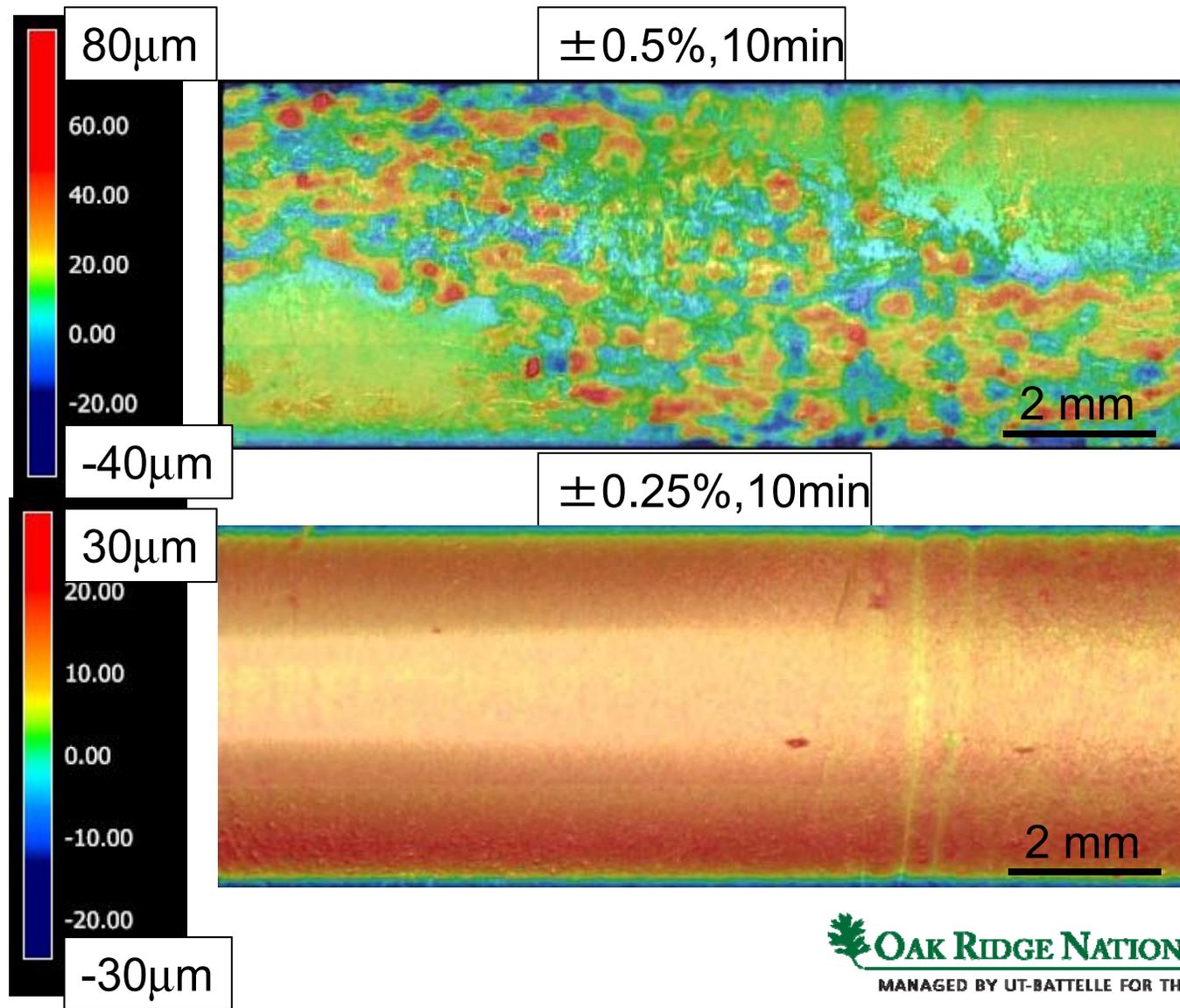
# Slow Evolution of "Steady Stress" for 10h Hold Time Test



# Thicker Oxide Scale for Tests Performed at $\pm 0.5\%$ , 10min Hold Time



# Higher Strain Leads to Oxide Scale Cracking and Re-oxidation

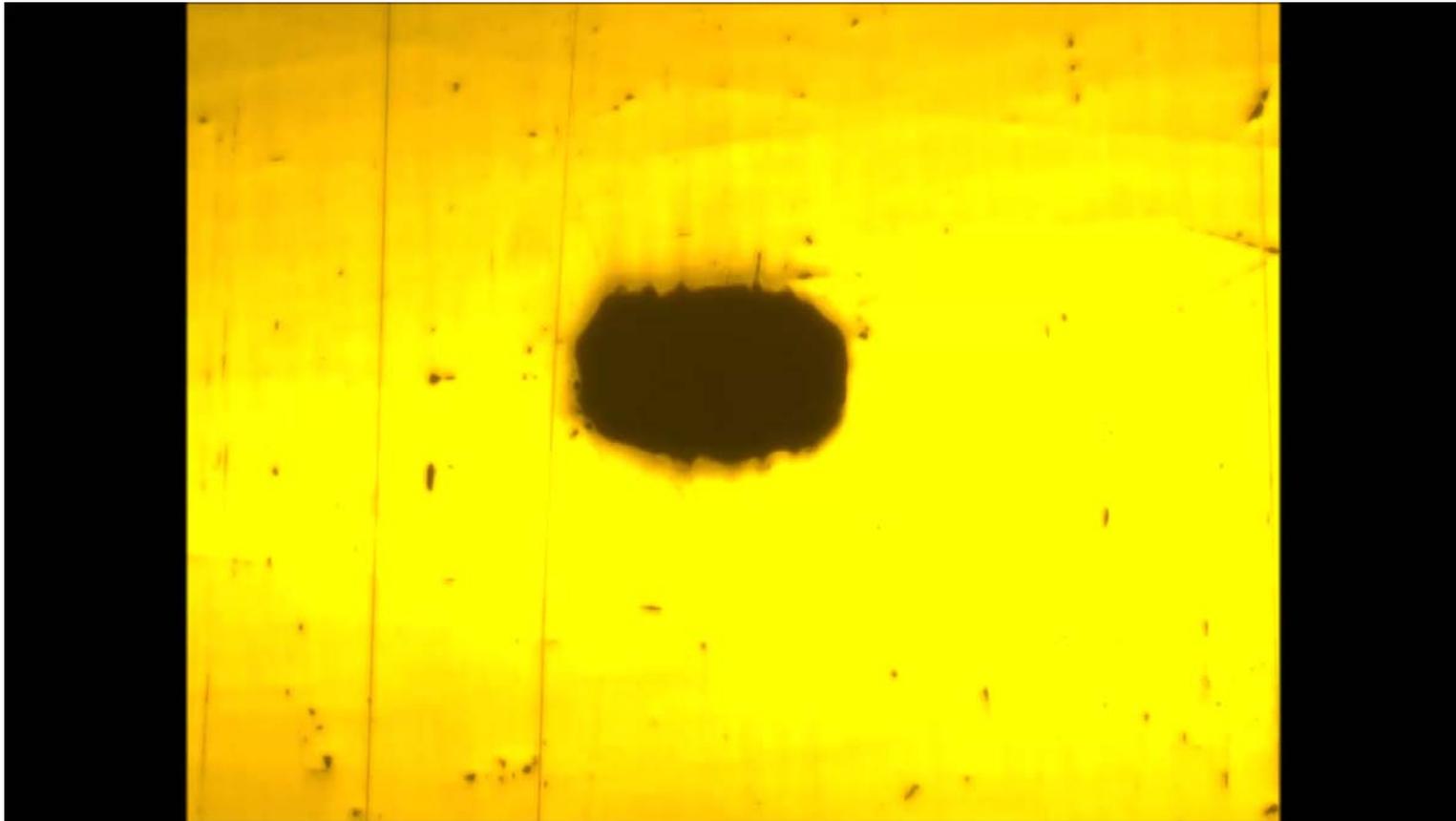


# New Set up to Study Microstructurally Small Crack Growth at High T°C

- Sumit Bahl's work (Indian Institute of Science)
- Slower propagation for small cracks
- Fournier et Al.(2008)
  - - Initiation: Tanaka and Mura model
  - - Propagation: Tomkins model
- Crack initiation at room temperature
- High cyclic Fatigue & Creep Fatigue Testing
- In Situ imaging of crack propagation
- Tests conducted at Room and 550°C

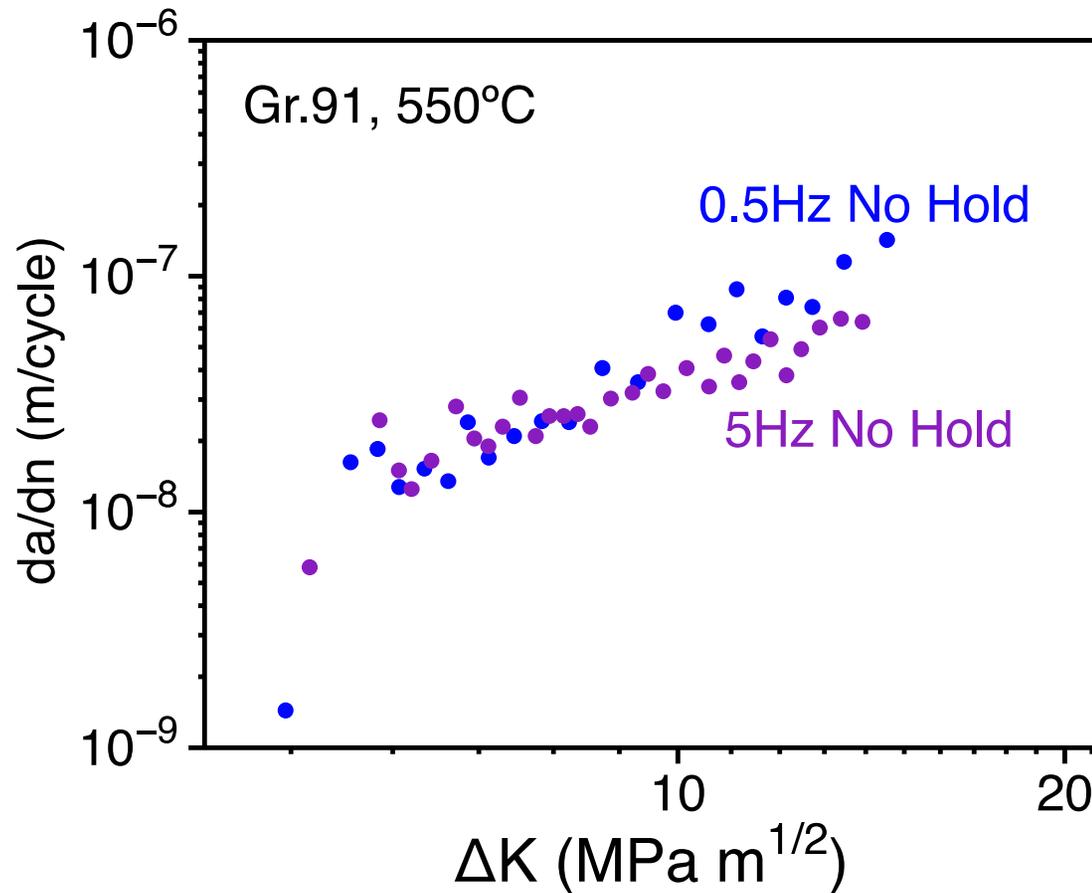


# Crack Growth Imaging at Room T°C

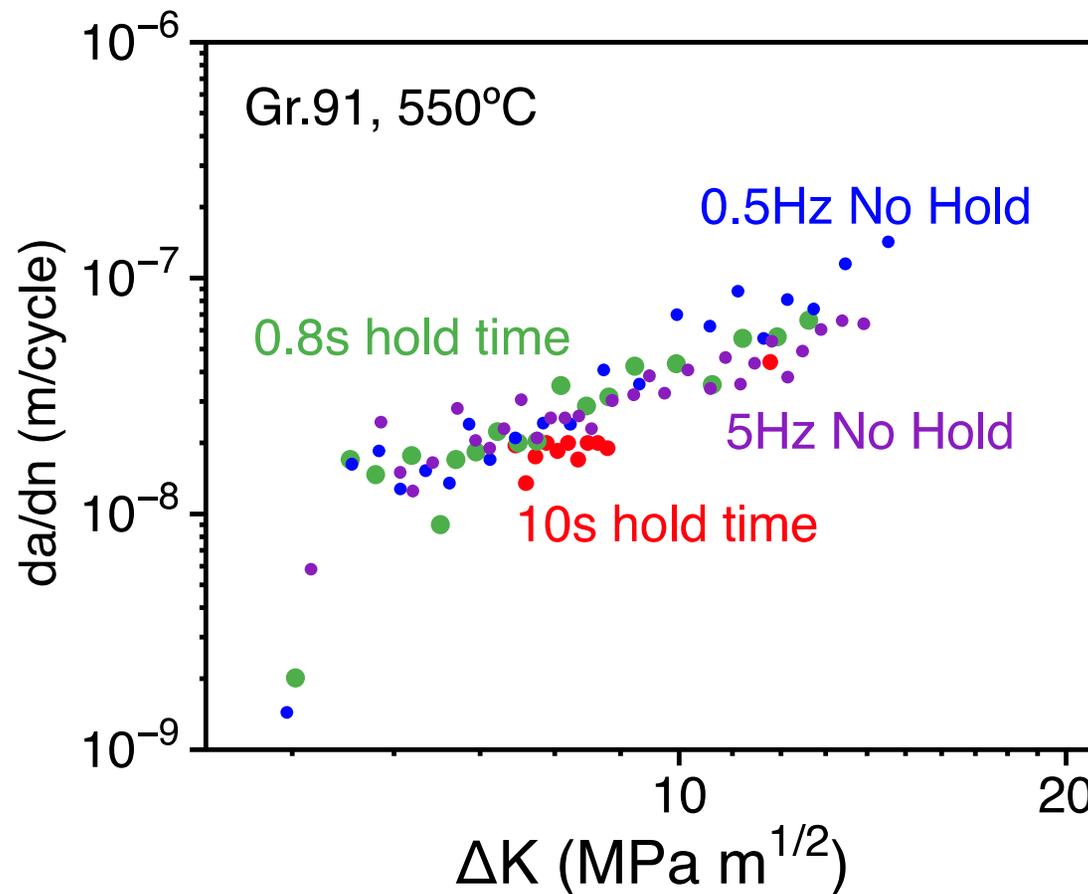


300μm

# No Frequency Effect on Crack Propagation



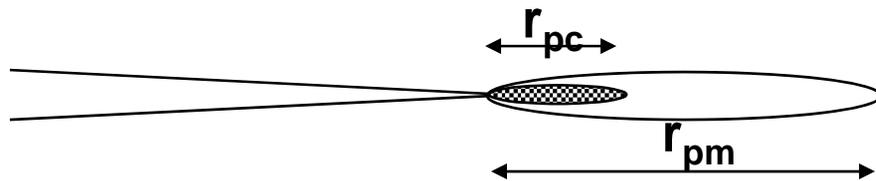
# No Effect of Hold Time on Crack Propagation



Test initiated with  
10min hold time

Results consistent with  
decrease of dislocation  
density at crack tip

# BCS\* Theory Crack Growth Characterization



$$r_{pm} = a \left[ \sec\left(\frac{\pi}{2} \frac{\sigma_{max}}{\sigma_{ys}}\right) - 1 \right]$$

**Monotonic Plastic Zone Size\***

Monotonic and Cyclic Plasticity are Related by Load Ratio

Substitute  $\sigma_{max} \rightarrow \sigma_{max} - \sigma_{min} = \sigma_{max}(1-R)$  and  $\sigma_{ys} \rightarrow 2\sigma_{ys}$

**Cyclic Plastic Zone Size**

$$r_{pc} = a \left[ \sec\left(\frac{\pi}{2} \frac{\sigma_{max}(1-R)}{2\sigma_{ys}}\right) - 1 \right] = a \left[ \sec\left(\frac{\pi}{4} \frac{\sigma_{max}(1-R)}{\sigma_{ys}}\right) - 1 \right]$$

$$R = -1, r_{pc} = r_{pm}; R > 0, r_{pc} < r_{pm}$$

Crack tip displacements (for distribution of edge dislocations at crack tip)\*\*

$$\phi_m = \frac{8\sigma_{ys}(1-\nu^2)a}{\pi E} \ln\left(\sec\left(\frac{\pi\sigma_{max}}{2\sigma_{ys}}\right)\right)$$

**Monotonic Crack Tip Displacement**

$$\phi_c = \frac{16\sigma_{ys}(1-\nu^2)a}{\pi E} \ln\left(\sec\left(\frac{\pi\sigma_{max}(1-R)}{4\sigma_{ys}}\right)\right)$$

**Cyclic Crack Tip Displacement**

\* Bilby BA, Cottrell AH, Swinden KH. Proc Royal Soc A 1963;272:304.

\*\* Weertman J. "Dislocations Based Fracture Mechanics"

# Small Fatigue Crack Growth Model

Number of cycles to failure → 
$$\Delta N = \frac{\phi_{cr}}{f\phi_c}$$

Crack extension at failure → 
$$\Delta a = \alpha \phi_m \sigma_{ys}$$

$$\frac{da}{dN} = \mu \phi_c \phi_m \sigma_{ys}$$

where 
$$\mu = \frac{f\alpha}{\phi_{cr}}$$

Crack-extension force on dislocations in monotonic plastic zone

$\phi_c$  – cyclic crack-tip displacement

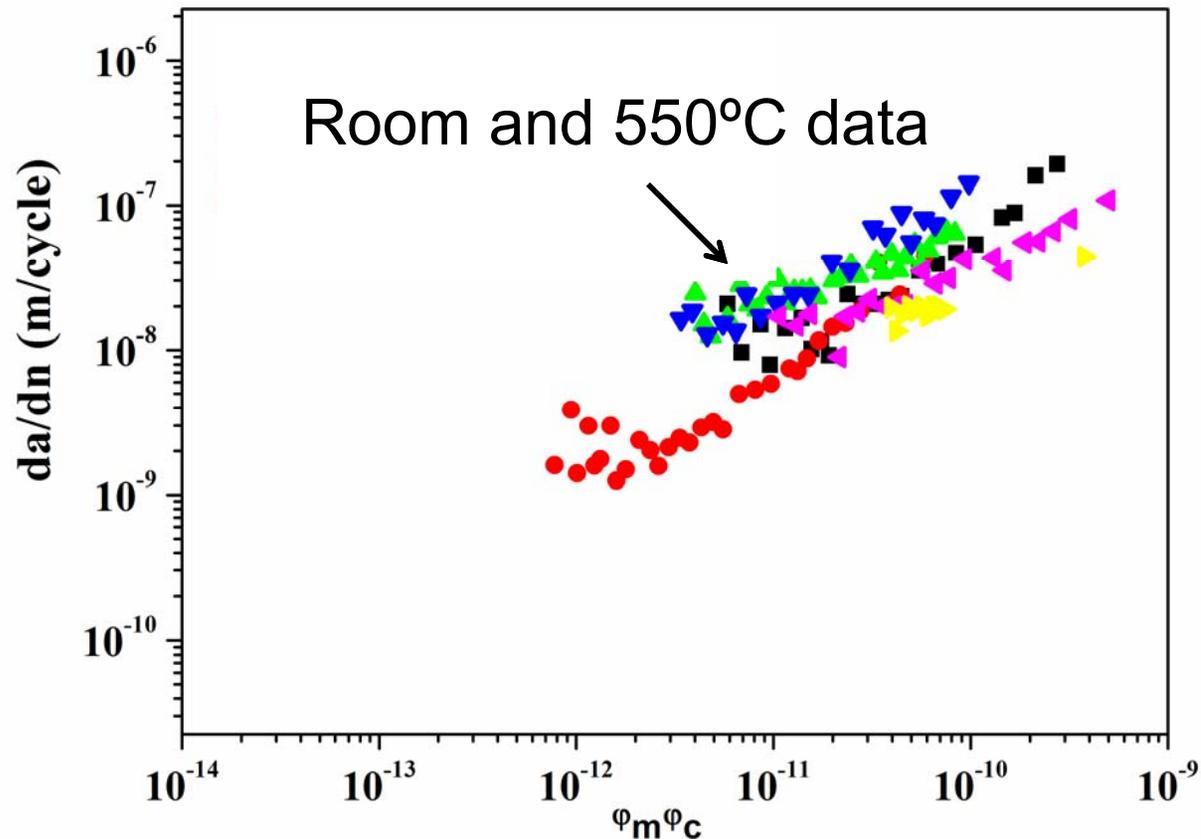
$\phi_m$  – monotonic crack-tip displacement

$f$  – slip irreversibility factor

$\phi_{cr}$  – critical opening displacement

$\alpha$  – geometric factor

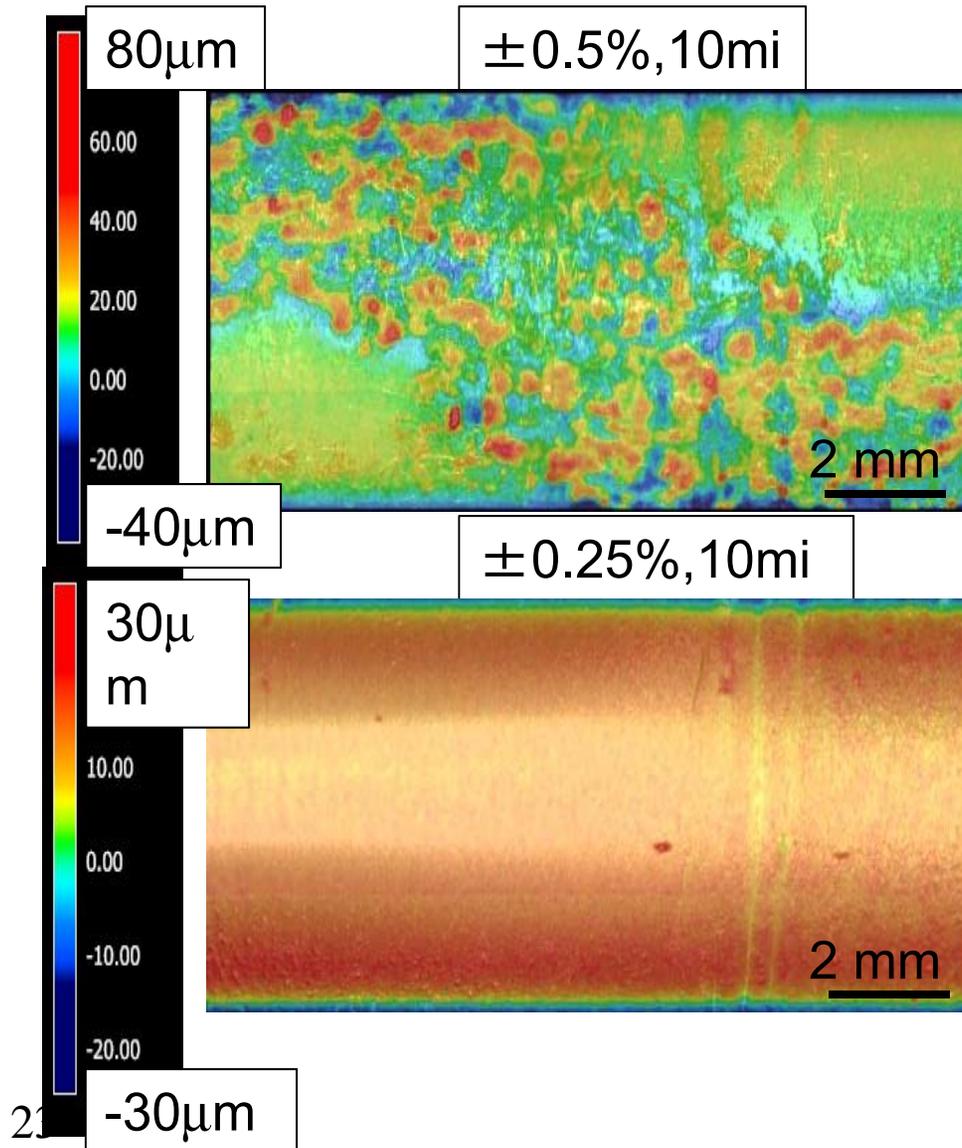
# Results at room and 550°C Consistent with Crack Growth Model



Tensile testing at different deformation rates

Microstructure characterization to evaluate dislocation density

# Creep-Fatigue-Oxidation Interaction in Steam?



In Air:

From protective scale to non-protective scale due to cracking and re-oxidation

In Steam:

Effect of strain on Non-protective scale?

# Martensitic Gr. 91 and fully ferritic 9Cr alloys creep tested at 650°C in air and steam

Alloys	Fe	Cr	Mo	Si	Mn	V	C
Gr.91-1	Bal.	8.31	0.9	0.13	0.34	0.26	0.08
Gr.91-2 (or 9Cr)	Bal.	8.61	0.89	0.11	0.27	0.21	0.08

## **Similar composition but different microstructures:**

-Gr.91-1 = standard commercial heat treatment

*Normalization: 1040°C and Tempering: 730-760°C*

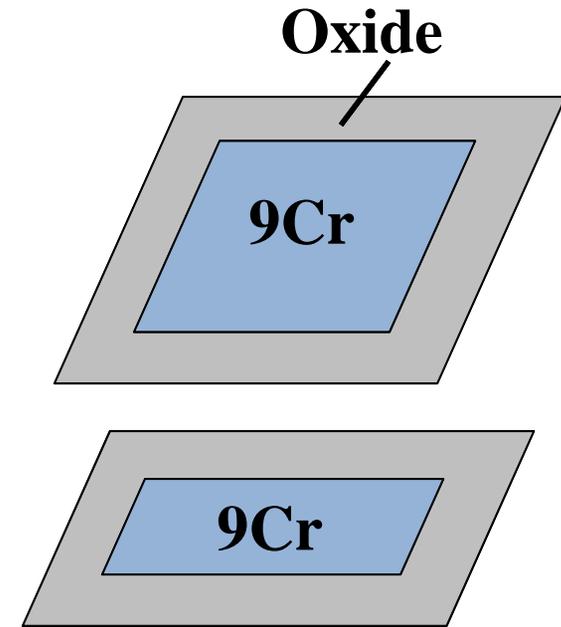
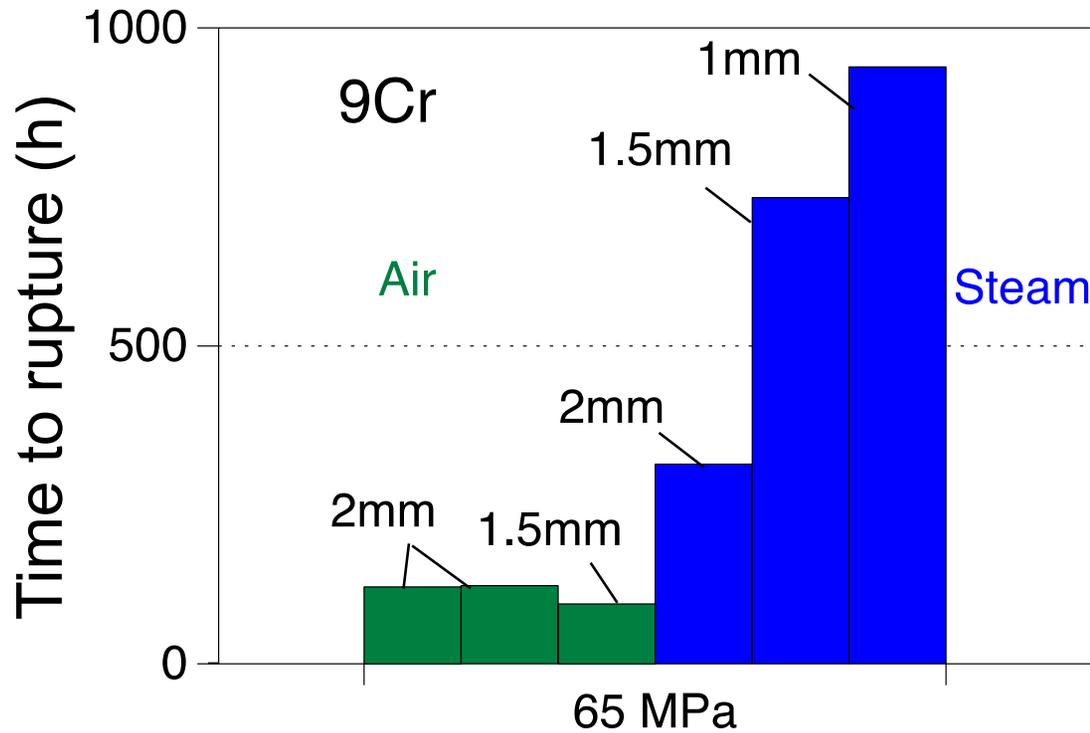
- Gr.91-2 = standard commercial heat treatment

*Normalization: 1080°C and Tempering: 760°C*

- 9Cr = Non heat treated material

*Fully ferritic as-fabricated microstructure*

# Effect of Load-Bearing Scale for Thin 9Cr Specimens



$$F = \sigma_{oxide} \frac{S_{oxide}}{S} + \sigma_{9Cr} \frac{S_{9Cr}}{S}$$

- Thinner specimen to increase the volume to surface ratio

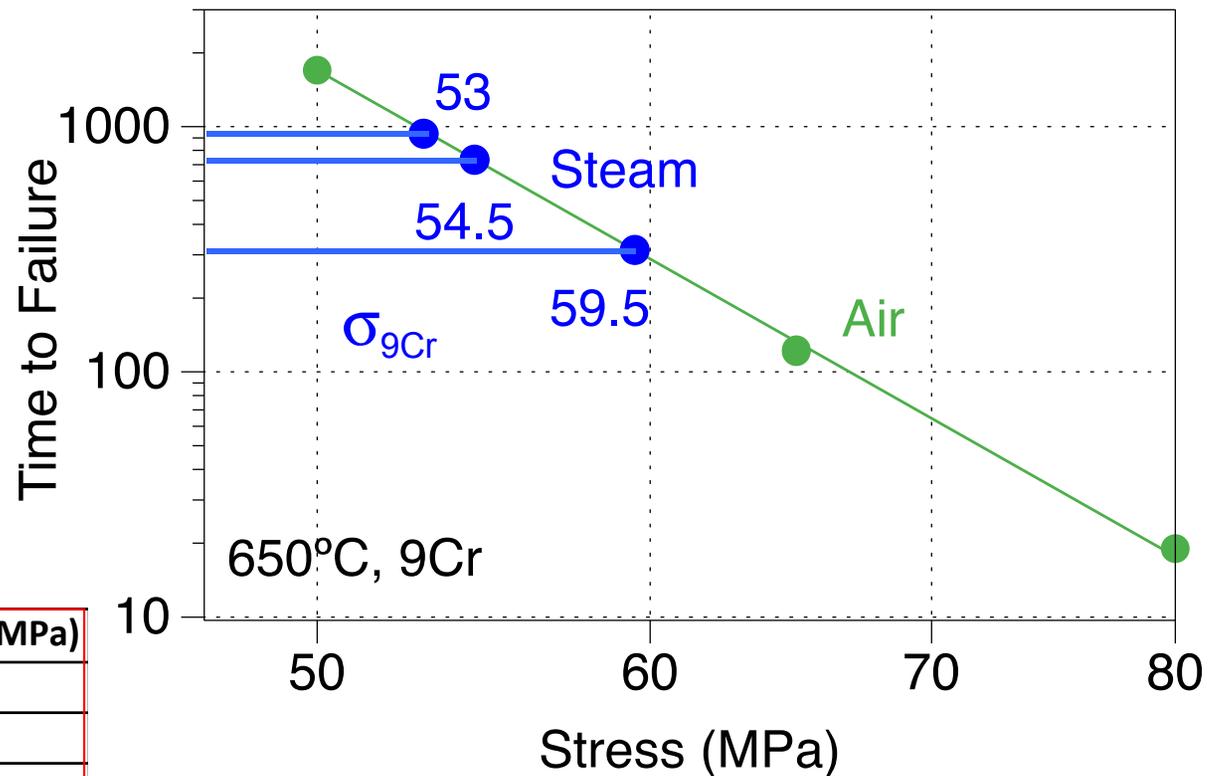
# Stress Calculation Is Very Consistent With Load Bearing Scale

$$F_{Applied} = \sigma_{oxide} \cdot S_{oxide} + \sigma_{9Cr} \cdot S_{9Cr}$$

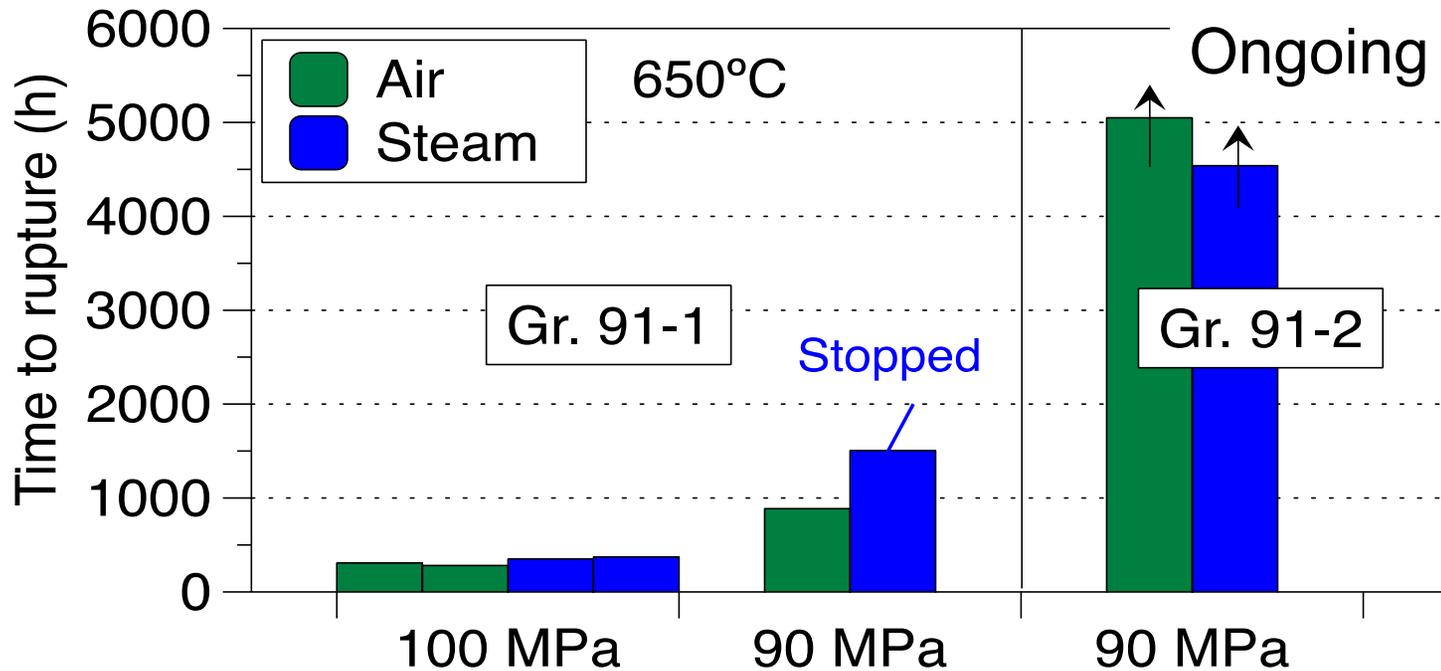
–  $\sigma_{9Cr}$  estimated from lifetime in steam

- Average oxide scale based on parabolic growth to calculate  $S_{oxide}$  and  $S_{9Cr}$

Thickness		
Specimen (mm)	Scale ( $\mu\text{m}$ )	$\sigma_{oxide}$ (MPa)
2	38	127
1.5	62	132
1	51	134

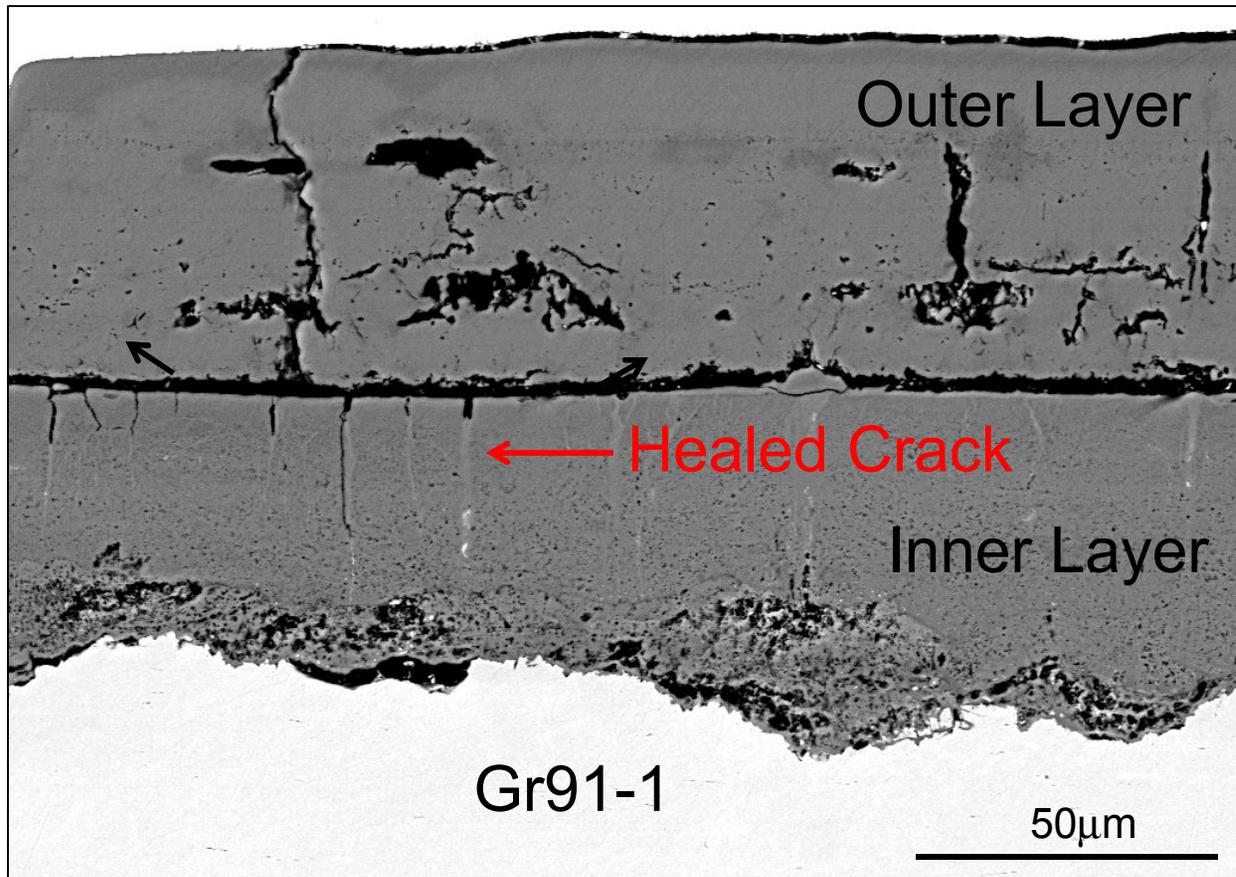


# No Effect or Small Increase of Lifetime in Steam vs Air for Gr.91 Alloys



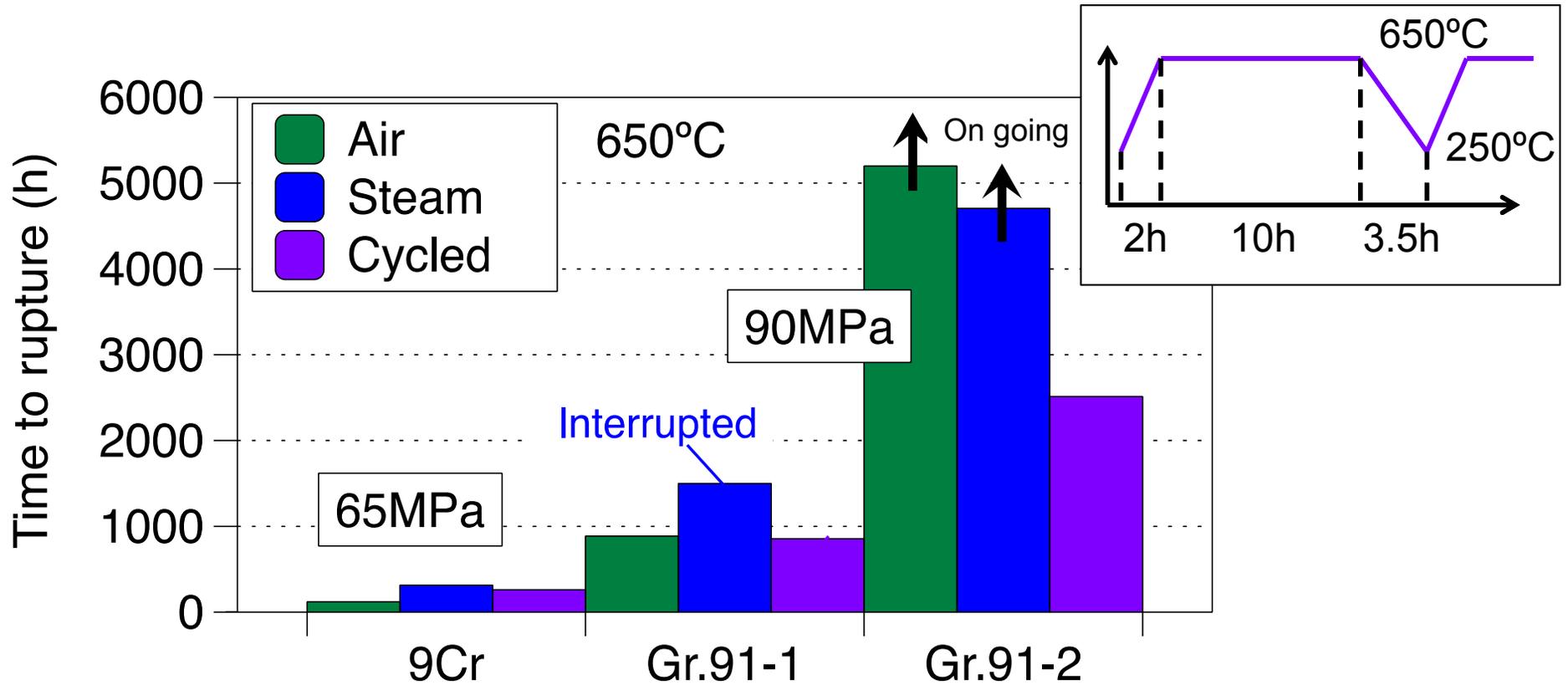
- Again consistent with Load-Bearing Scale compensating for metal loss

# Healed cracks lead to a Continuous Bearing Inner Oxide scale

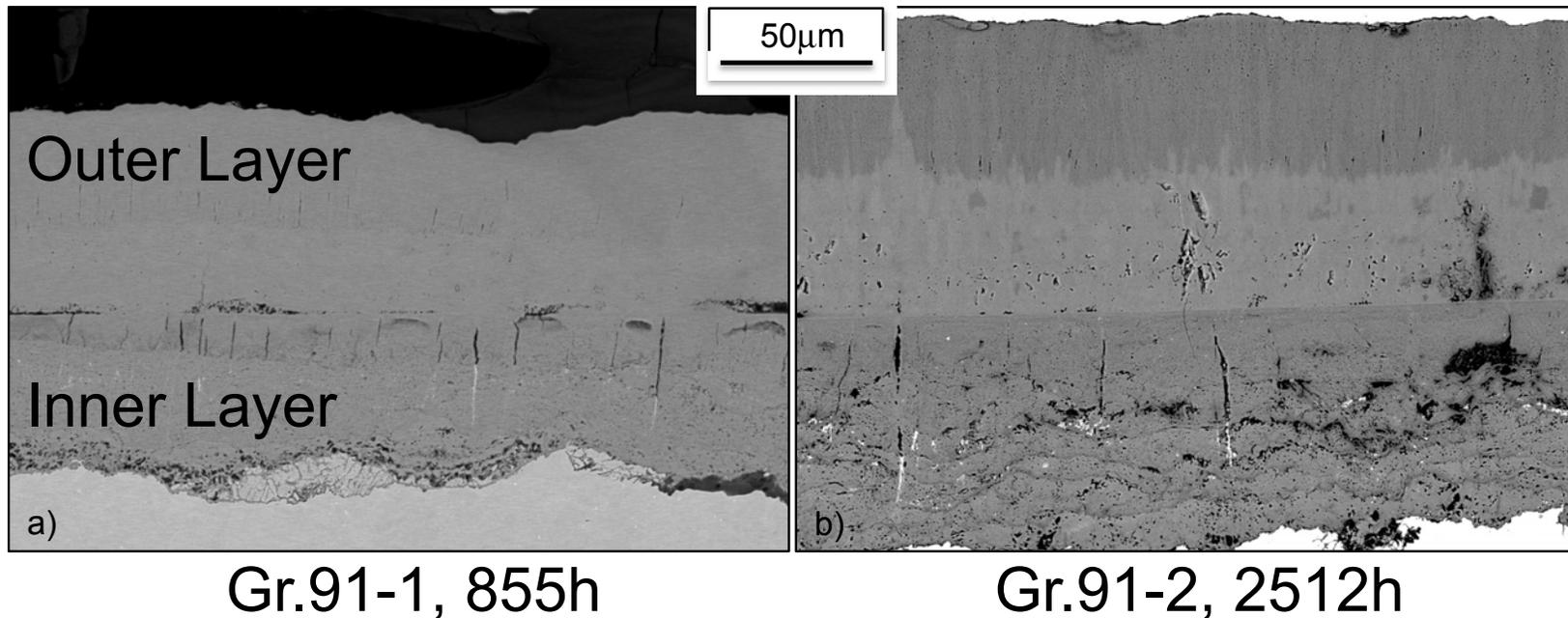


*Gr91-1, Steam, 350h, 100MPa*

# Decrease of Lifetime in Steam due to Thermal Cycling



# Similar Oxide Scale Microstructure for Specimens Thermally Cycled



- Local cracking of the scale leads to sudden increase of stress and rupture?
- Ongoing cyclic air test to verify Gr.91 microstructure is not affected by thermal cycling

# Future Activities

- Conduct microstructure characterization of cyclic creep, creep fatigue and fatigue crack growth specimens
  - Sub grain coarsening
  - Decrease of dislocation density
  - Cavity formation
- Continue long-term tests
- Conduct fatigue and creep-fatigue testing in steam.
  - Design is Ready
  - Effect of cyclic loading on a thick non protective scale?

# Acknowledgements

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