

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

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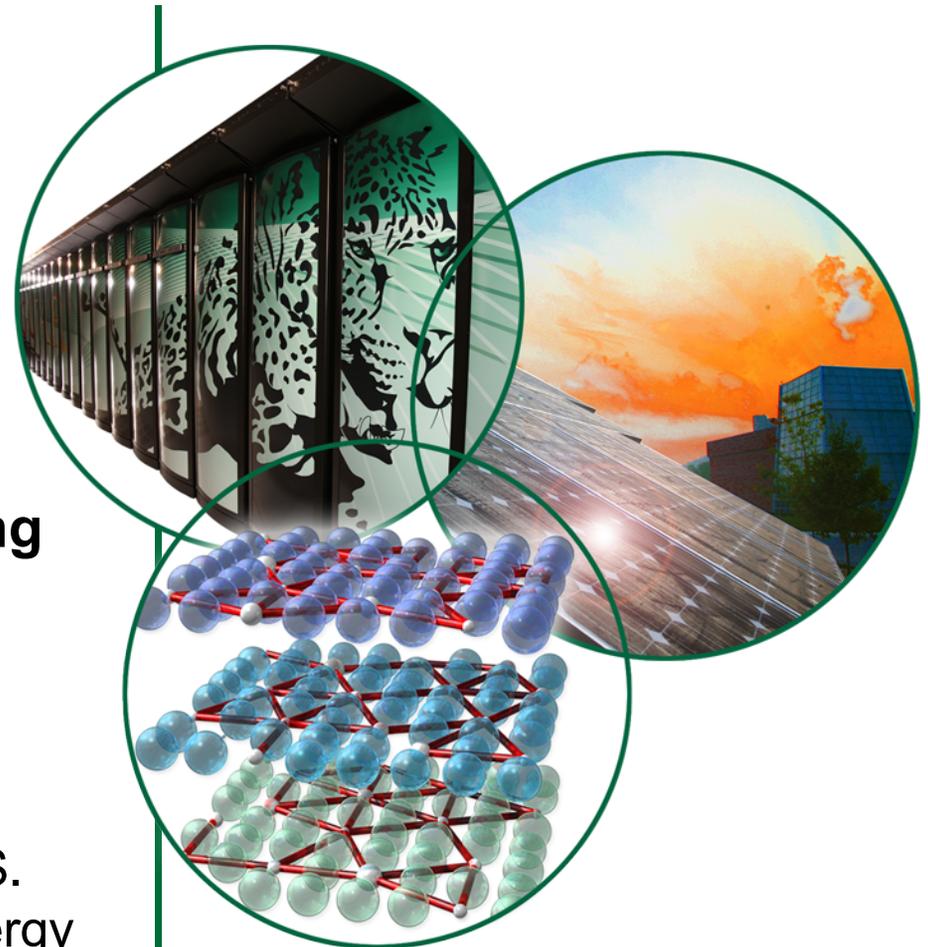
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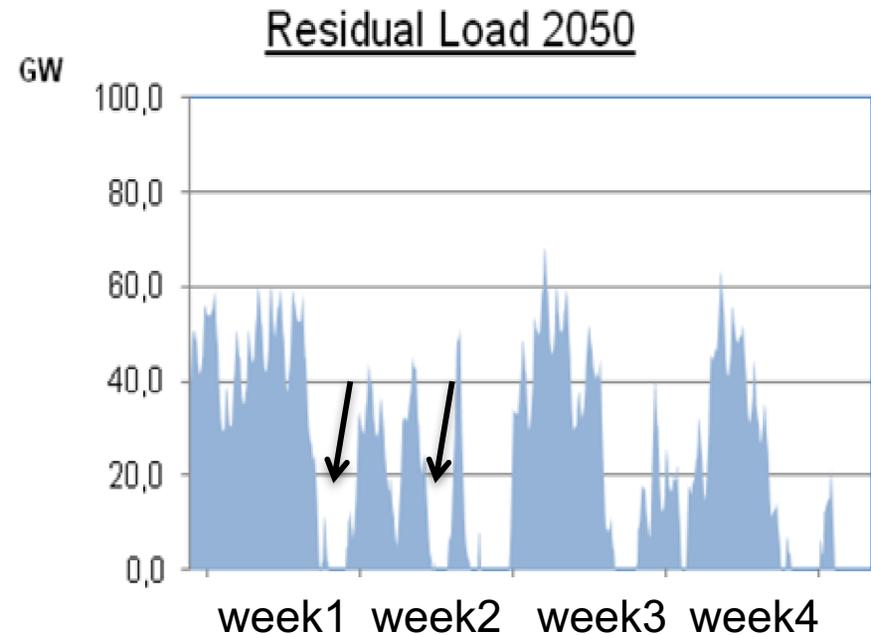
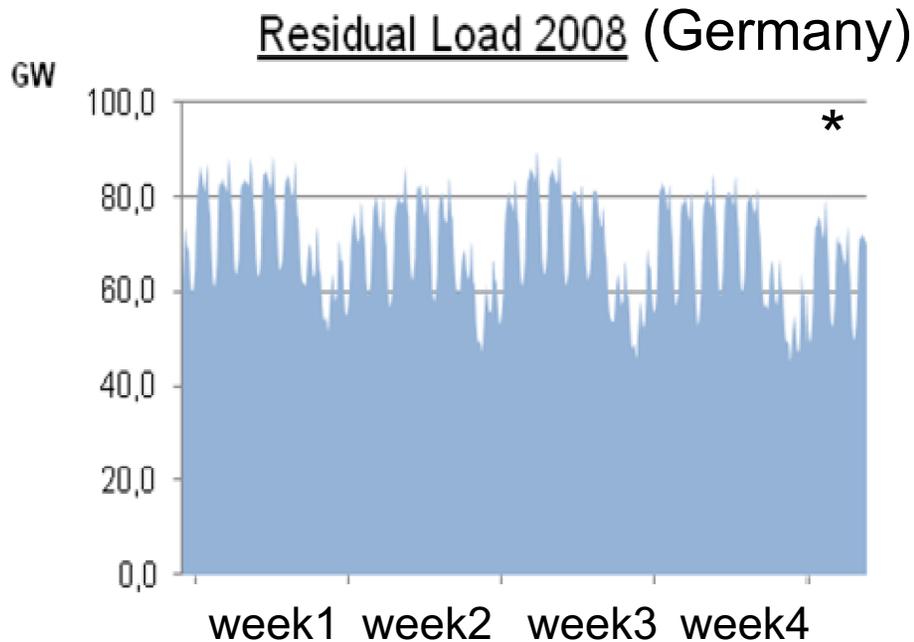
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# Projects Goals & Objectives

- Long term creep fatigue testing and lifetime modeling
- Interactions among creep fatigue and oxidation
- Study of microstructurally small cracks under creep-fatigue loading
  - Submit an open-literature paper on the development and propagation of microstructurally small cracks **03/31/17 met**
  - Perform at least three strain-controlled creep-fatigue tests in steam **02/15/17 met**
  - Develop a creep-fatigue lifetime model in air based on long term data + small cracks **06/31/17 and steam In progress**
  - ICME scheme based on the lifetime models developed to accelerate the design of creep-fatigue resistant alloys **In progress**
  - Conduct at least three thermo-mechanical tests **09/31/17**

# Power Plants Will Need to be Capable of Flexible Operation

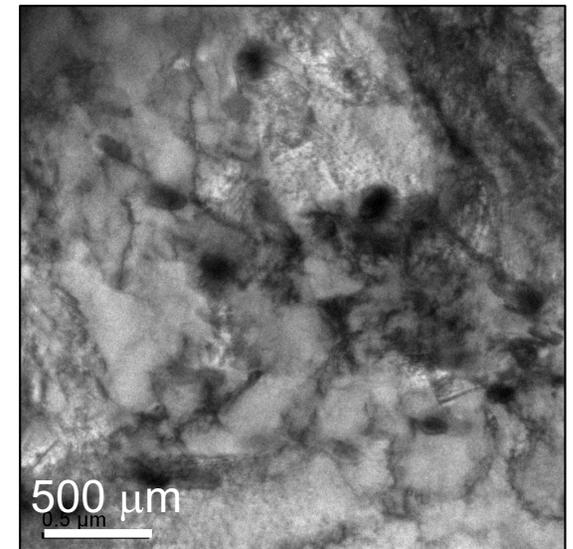
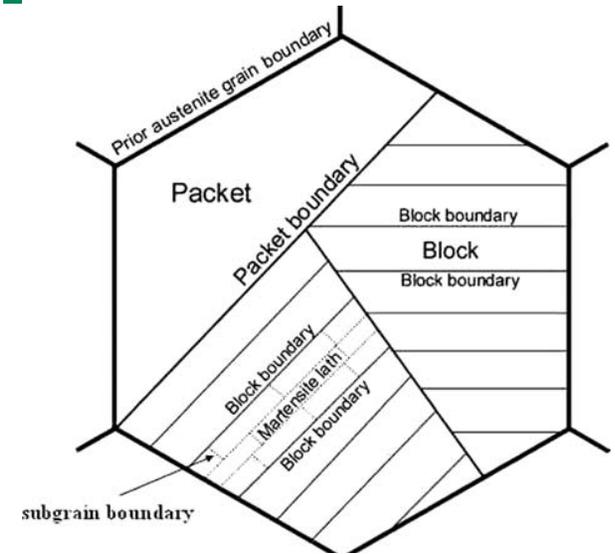


- Frequent (~daily) load cycling will result in significant creep-fatigue interaction

# Microstructural Creep Damage for Gr.91 9Cr-1Mo Steel

- Subgrain coarsening 1-5
- Decrease of Dislocation Density 1-5
- Particle coarsening (MX,  $M_{23}C_6$ ) 1-5
- Cavitation at GB 4-5
- Lave/Z phase formation 3
- Depletion of solid-solution elements 1
- Oxidation?

1 Semba et al. MHT (2008), 2 Orugandi et al. Acta Met (2011), 3 Sawada et al. MSE (2011), 4 Gaffard et al. Int jour Fat (2011), 5 Yadav et al. MSE (2016)



# Interaction of Creep & Fatigue Damages

- Sub grain coarsening / Dislocation density decrease.

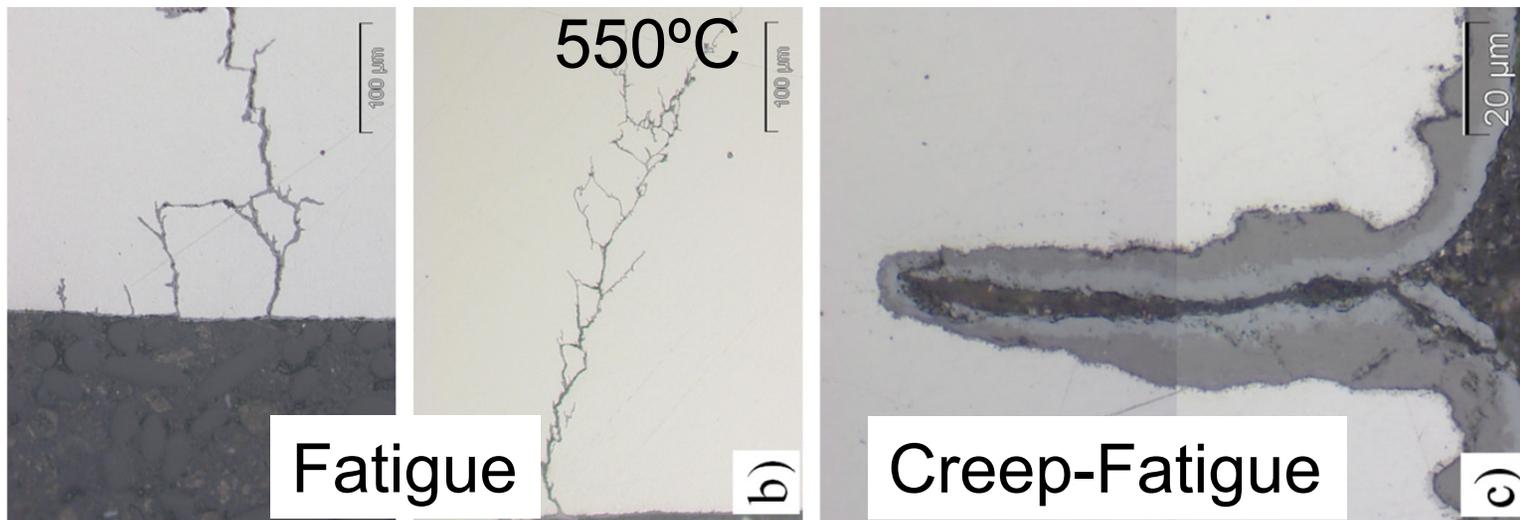
Creep performance significantly affected by strain cycling\*

- Effect of oxidation on crack initiation during Creep-Fatigue

Fast initiation due to cracking of the scale and propagation in the alloy\*

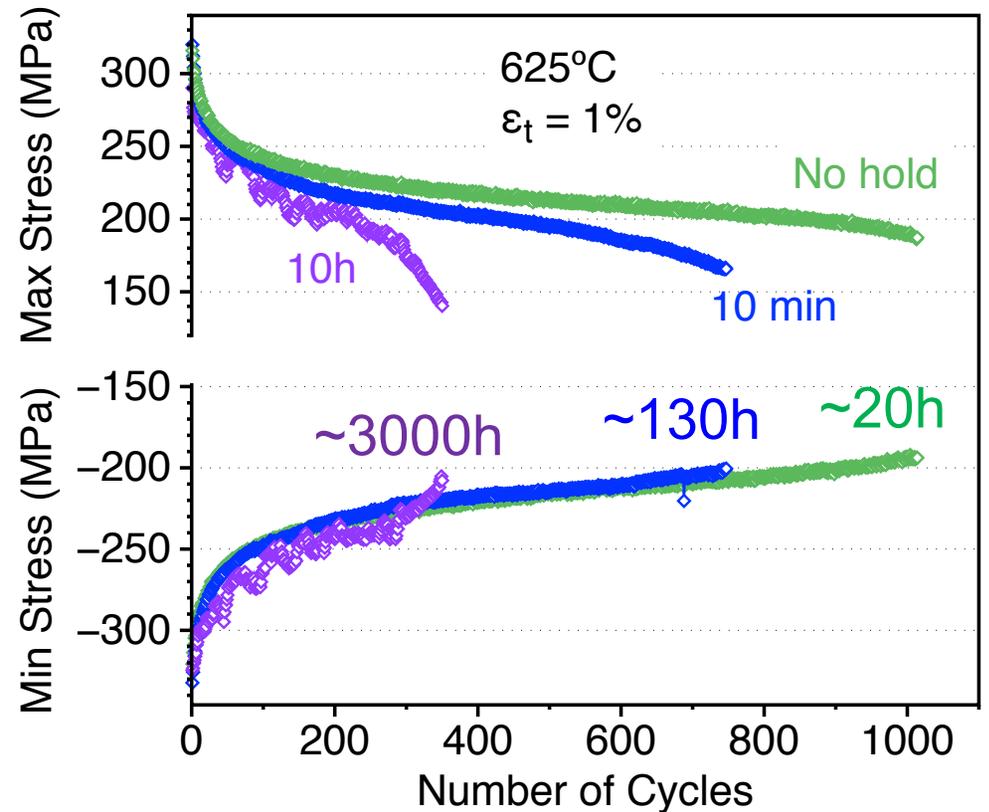
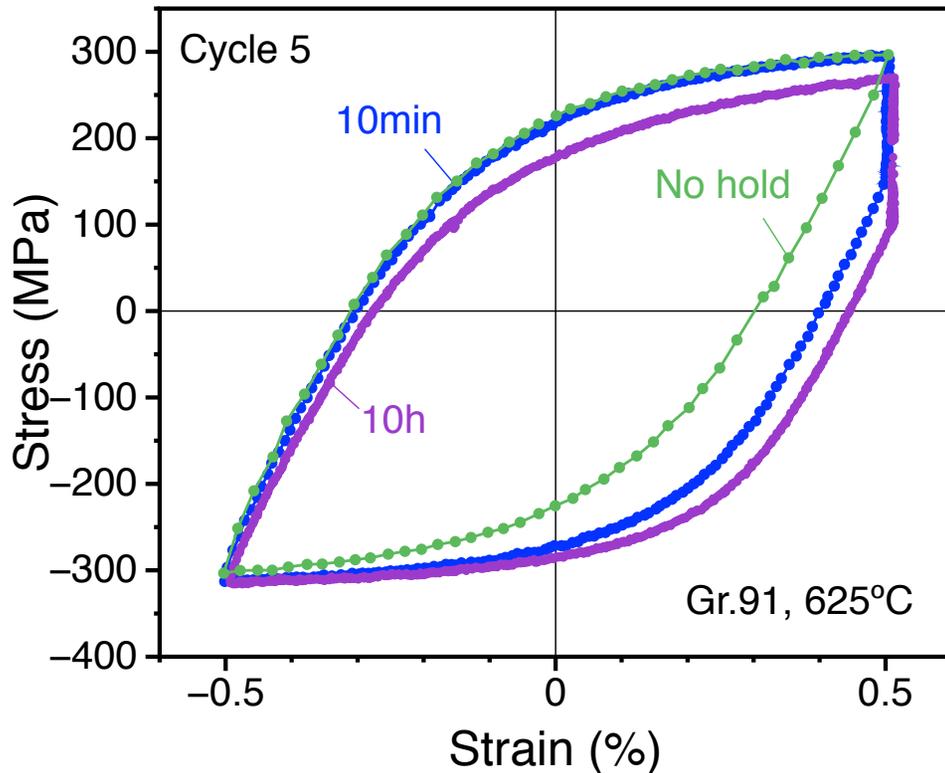
Much thicker non protective scale in air sue to cracking

- No obvious effect of oxidation/hold time on crack propagation?



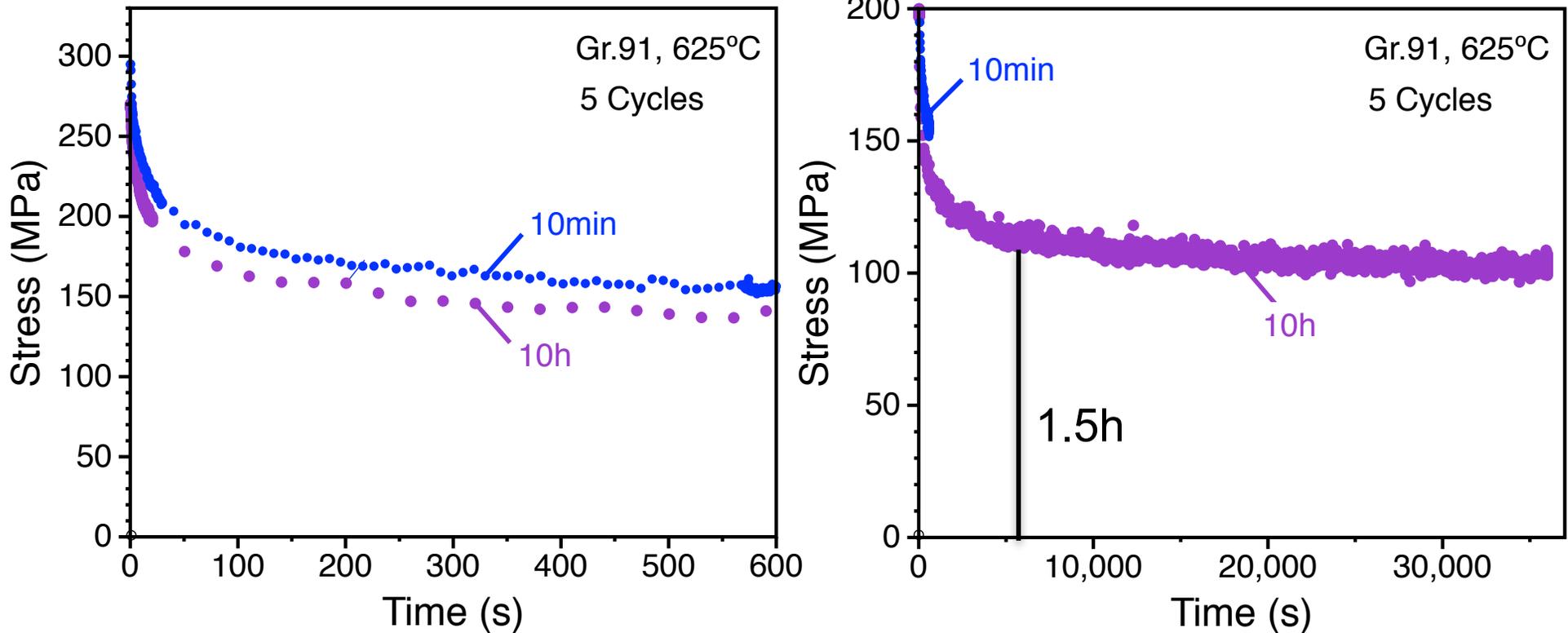
\*Fournier et al., Int. J. Fatigue 2008, M&M Trans A 2009

# Decrease of $N_f$ with Hold Time for Creep-fatigue Tests at 625°C, $\pm 0.5\%$



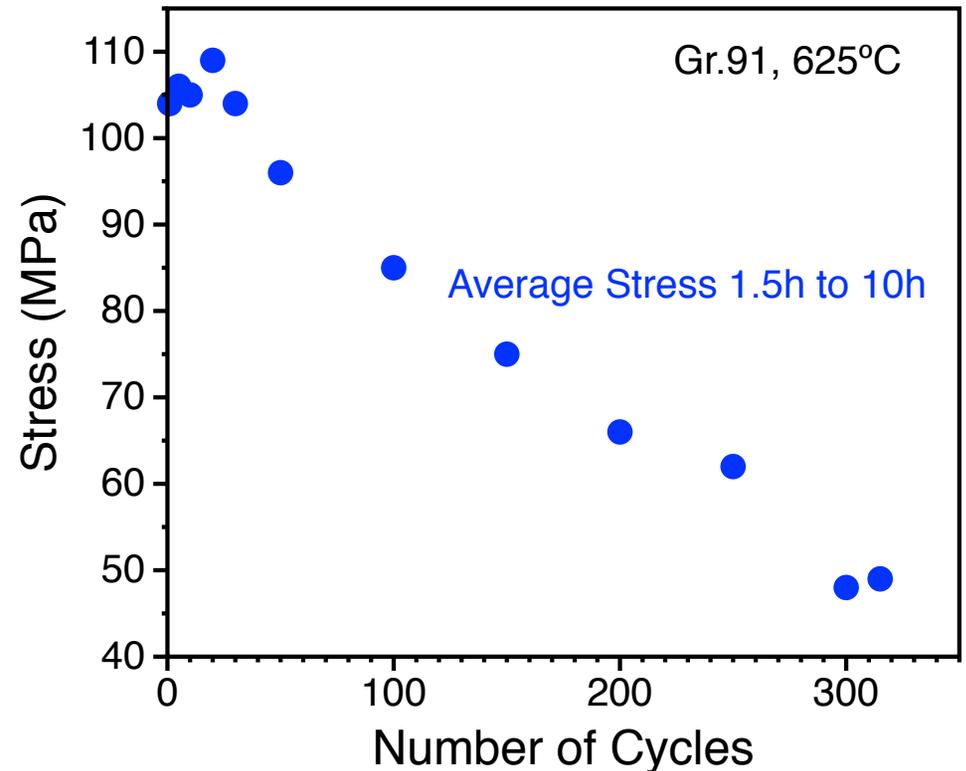
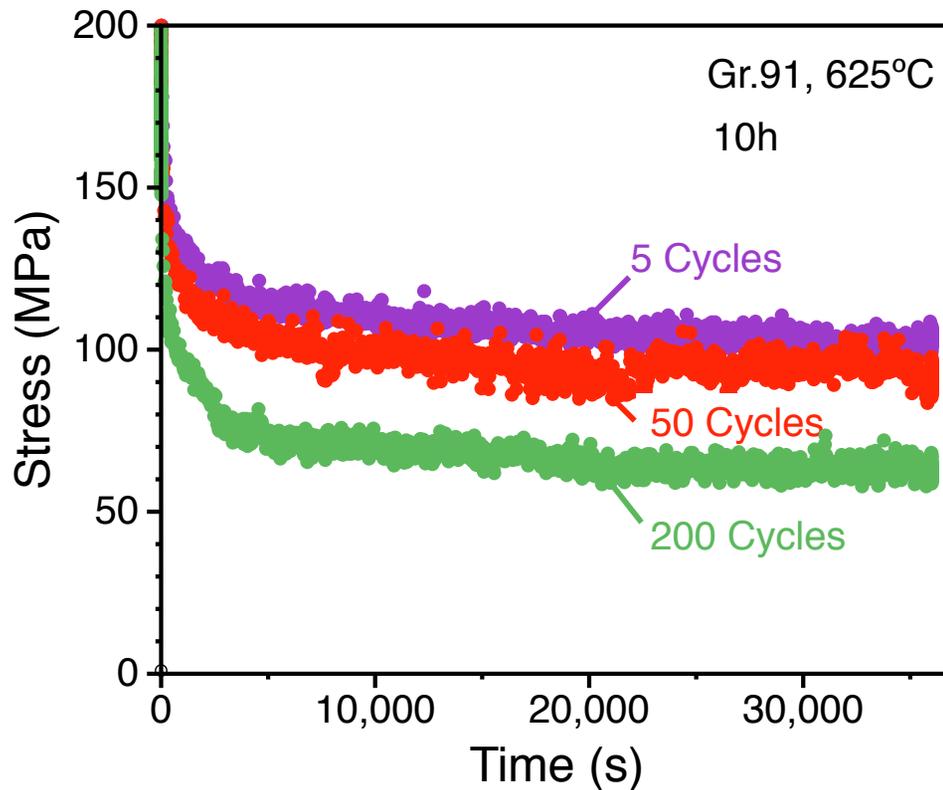
- Limited effect of hold time on softening rate
- Significant decrease of number of cycles to failure with longer hold time (>3000h test)

# Significant Stress Relaxation During 10h Hold Time



- Need ~ 1.5h to reach a nearly steady state stress
- Creep lifetime at 625°C, 100MPa ~5000h

# Linear Decrease of “Steady Stress” for 10h Hold Time Test After ~30 cycles



Significant effect of cycling on alloy creep behavior

# Thicker Oxide Scale for Tests in Air With $\pm 0.5\%$ , 10min and 10h Hold Time

$\pm 0.25\%$ , No hold  
50h

$\pm 0.25\%$ , 10min  
440h

$\pm 0.5\%$ , No hold  
20h

$\pm 0.5\%$ , 10min  
130h

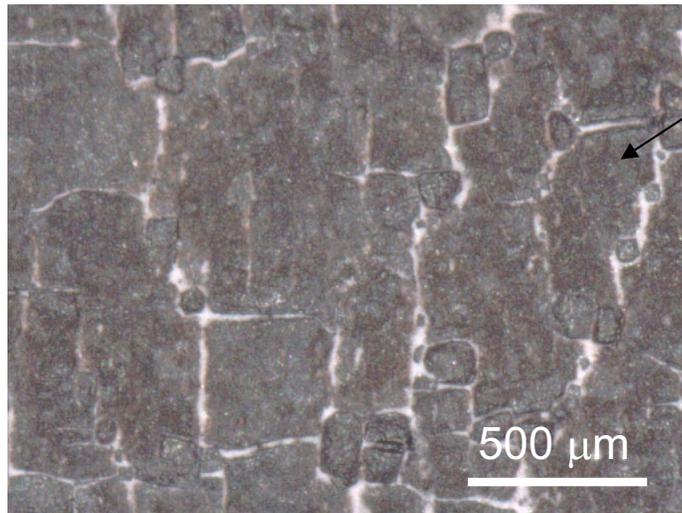
$\pm 0.5\%$ , 10h  
3000h

Local oxidation

Buckling

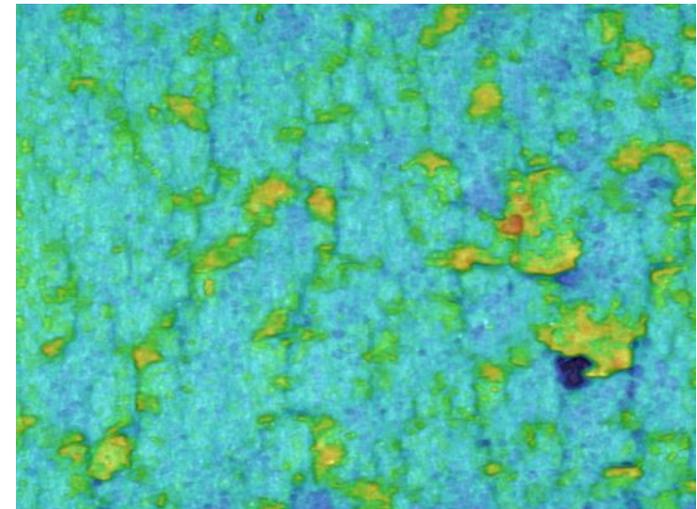
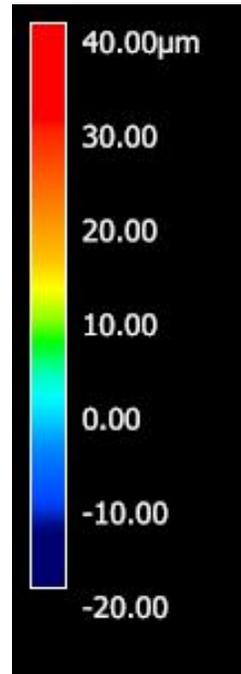
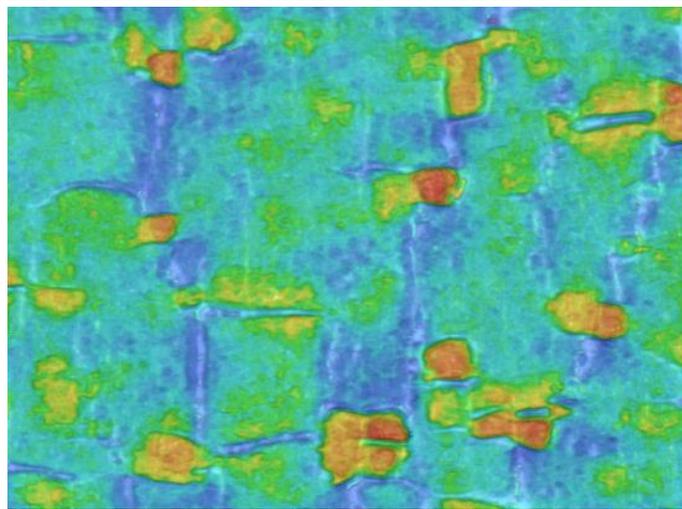
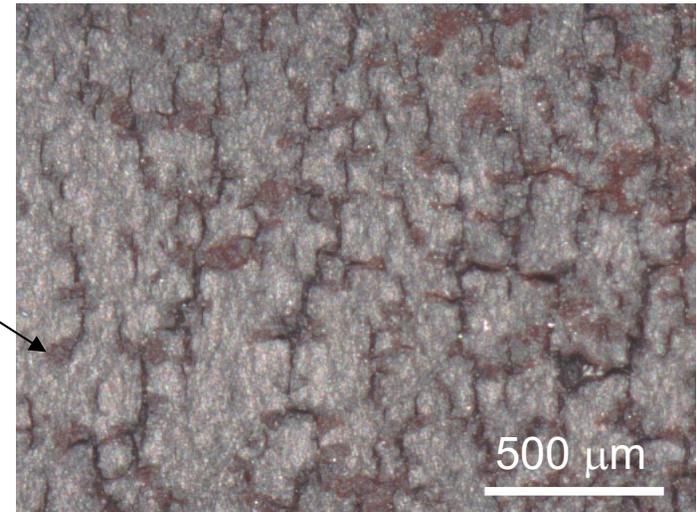


# Crack Pattern at 10min & 10h specimen surface. Thicker scale for 10min test

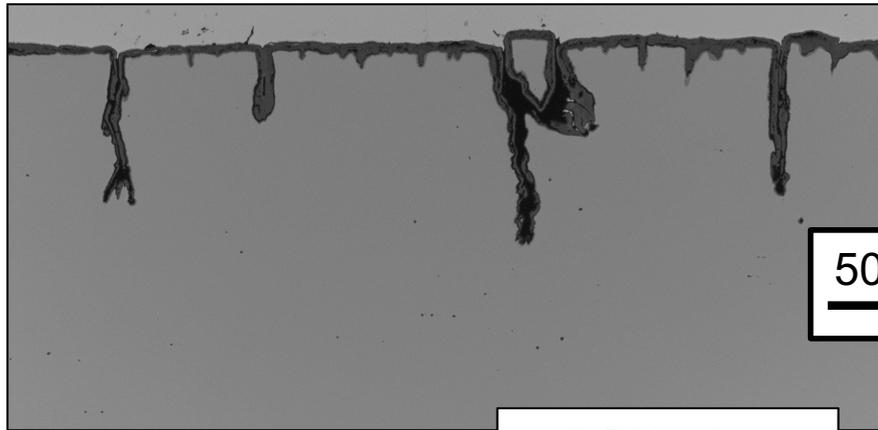


$\pm 0.5\%$ , 10min

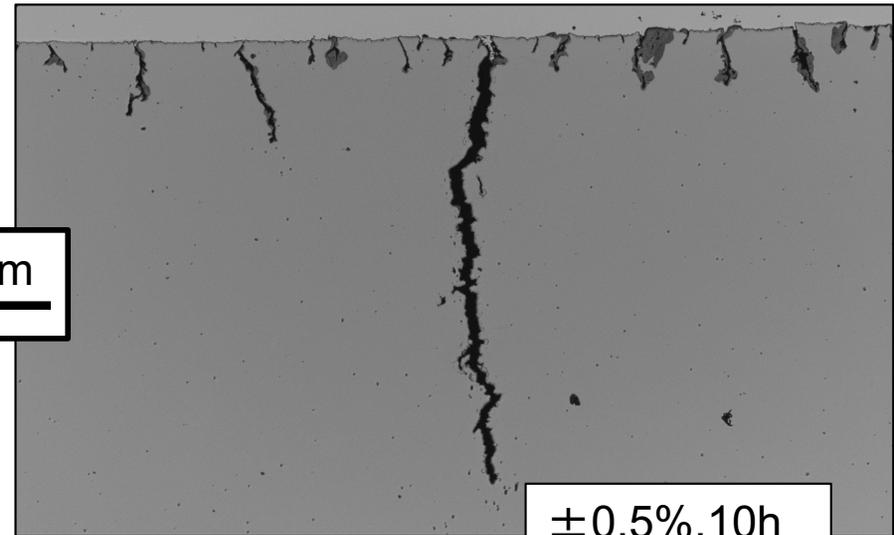
$\pm 0.5\%$ , 10h



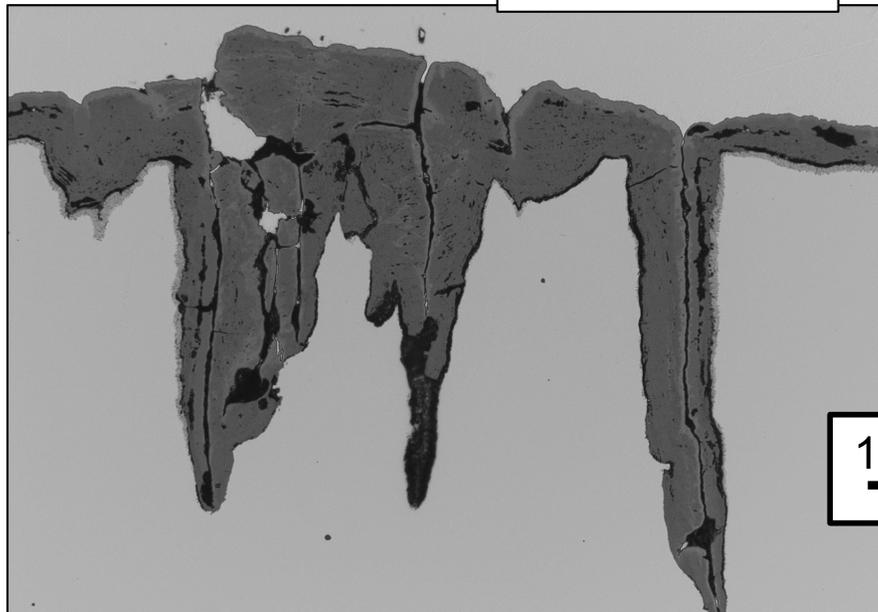
# Numerous Oxidized Cracks for 10min and 10h Hold Time Tests



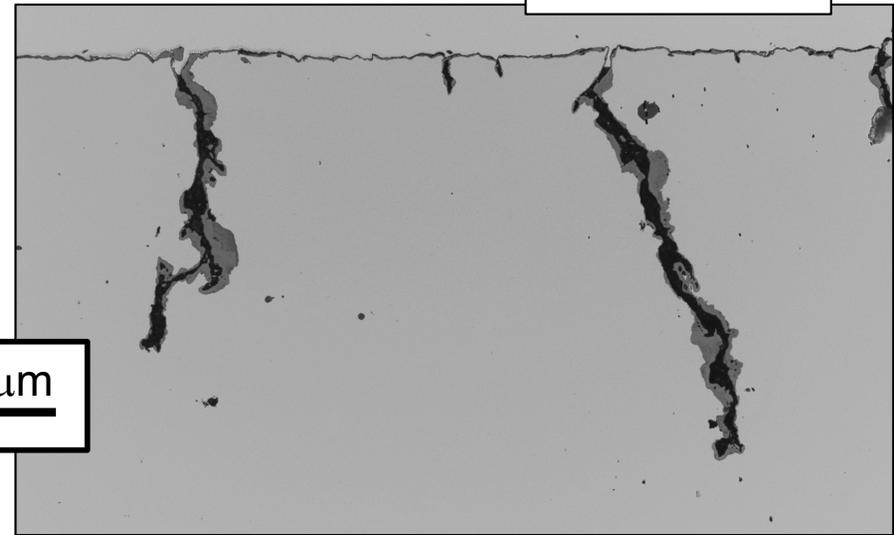
±0.5%, 10min



±0.5%, 10h

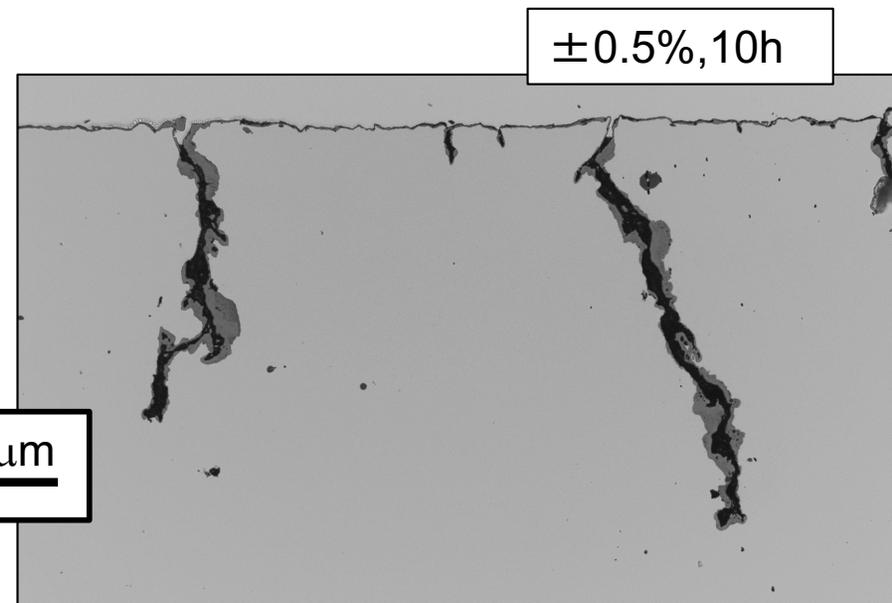
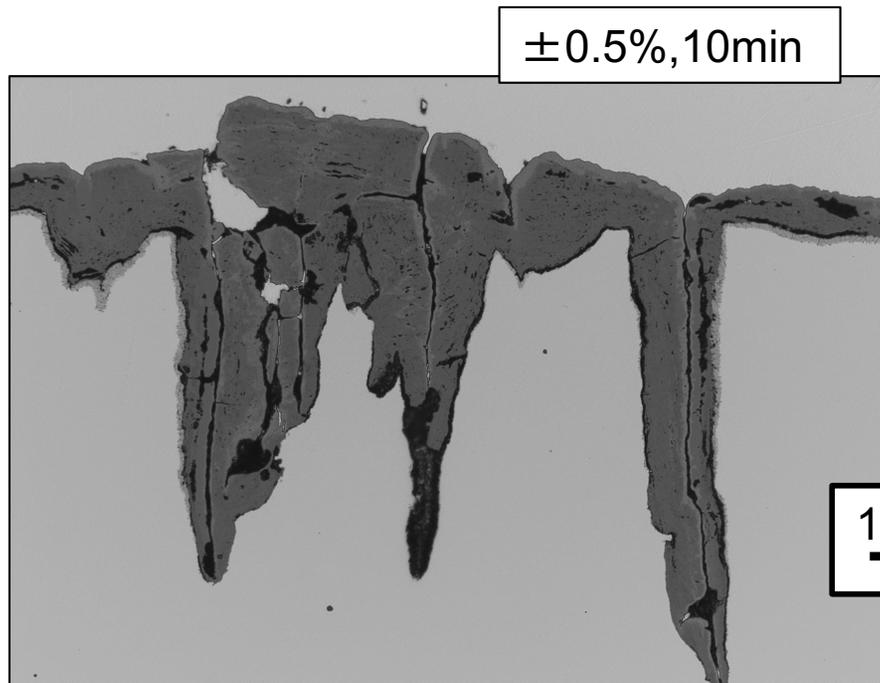
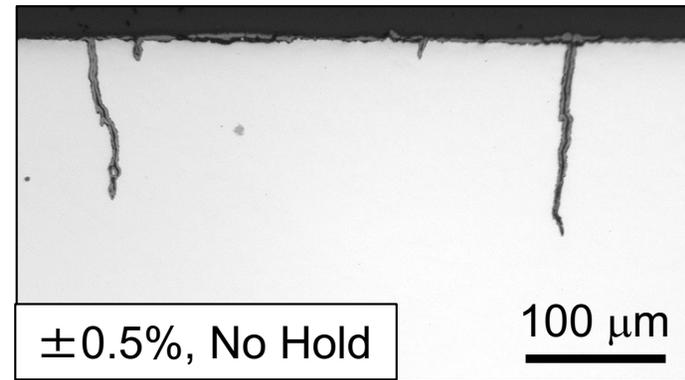


100 μm

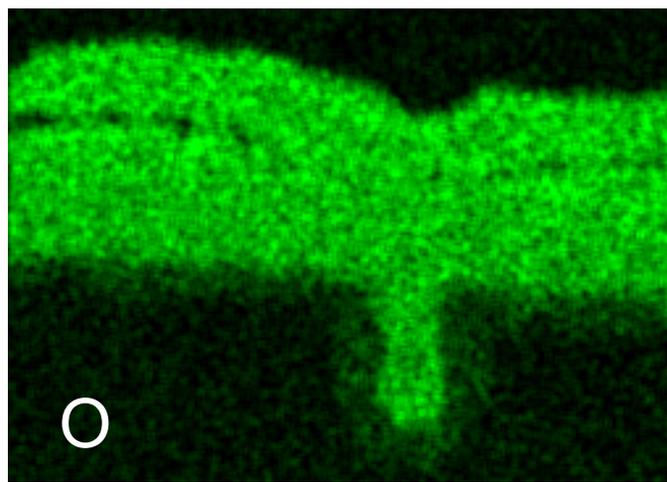
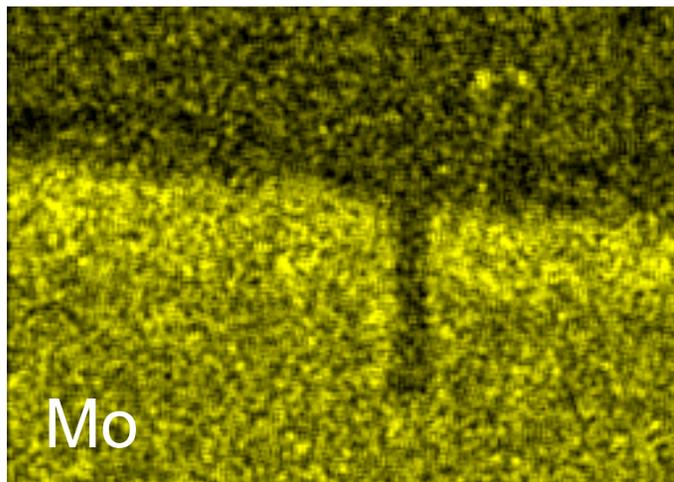
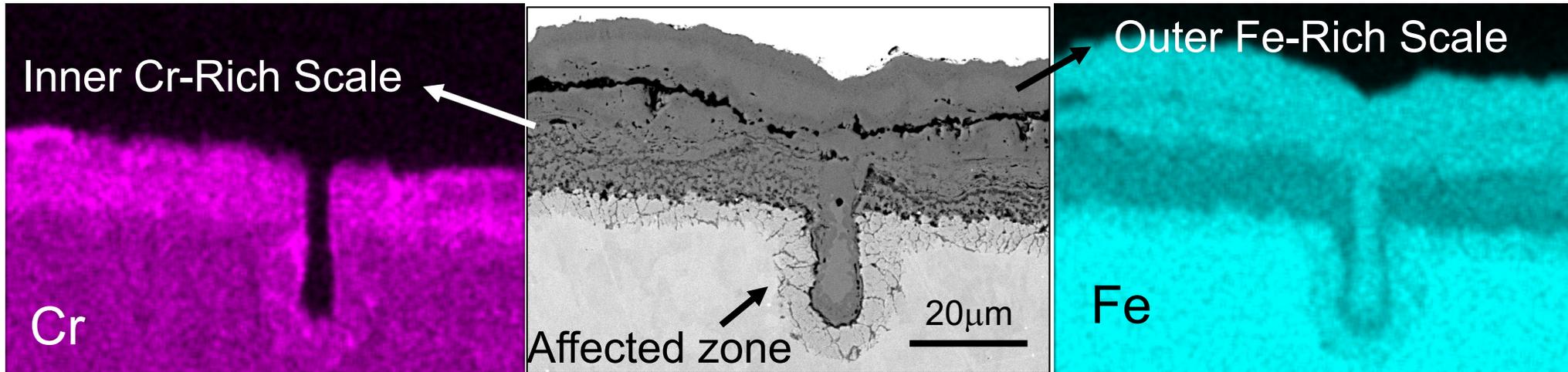


# Thicker Oxide Scale for 10min Hold Time Test

Few Localized Oxidized Cracks for Test without Hold

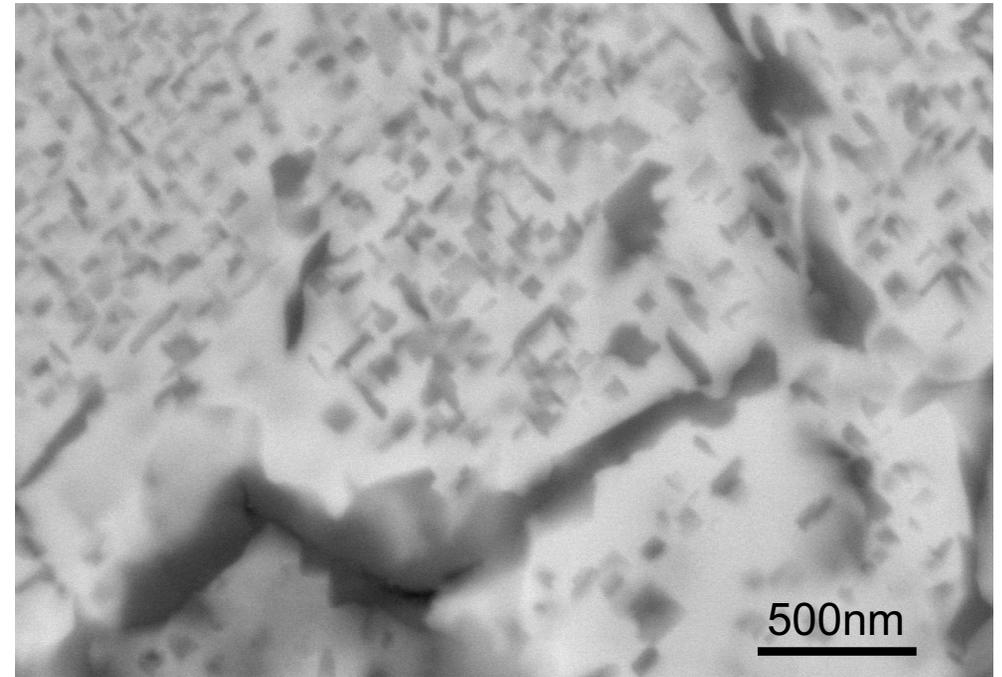
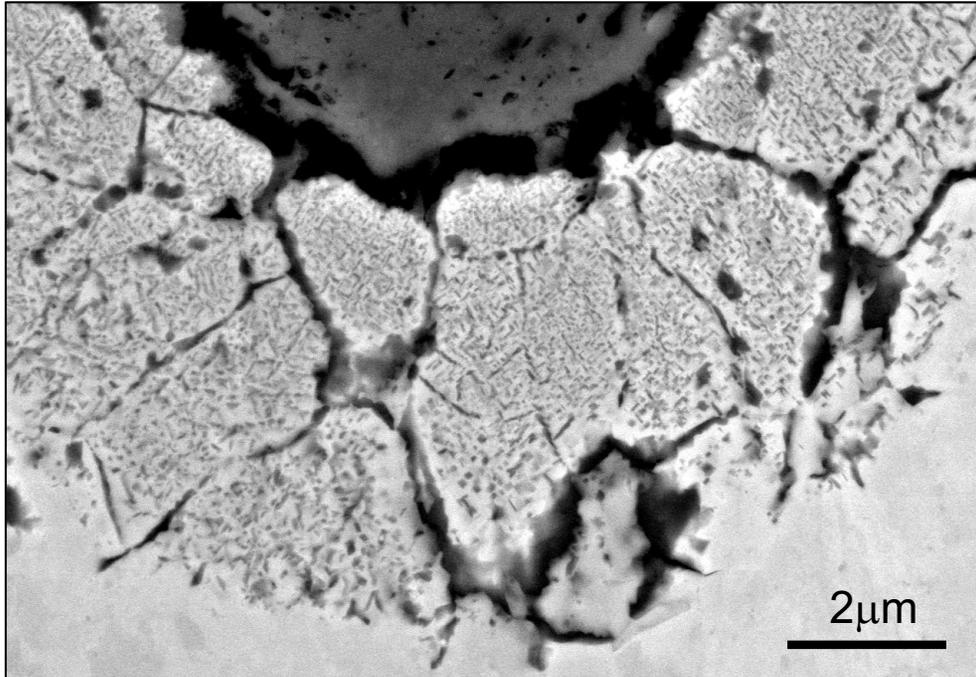


# Multi-Layer Oxide Scale for 10min Hold Time Test



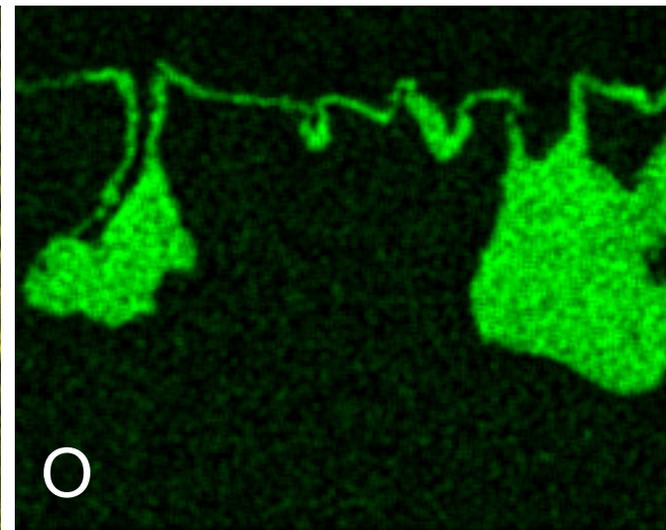
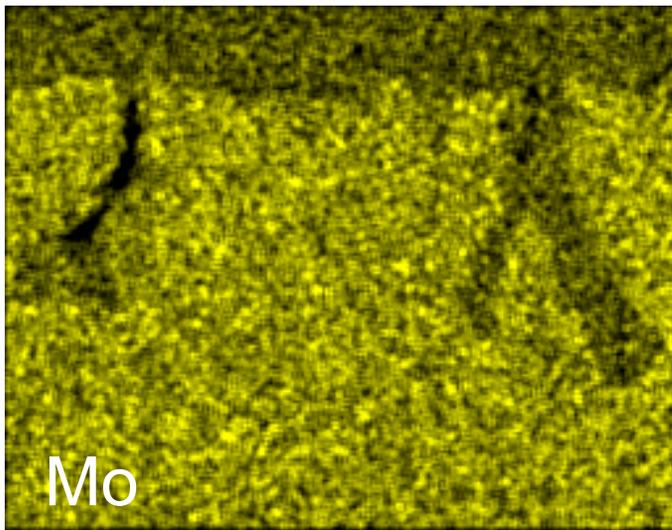
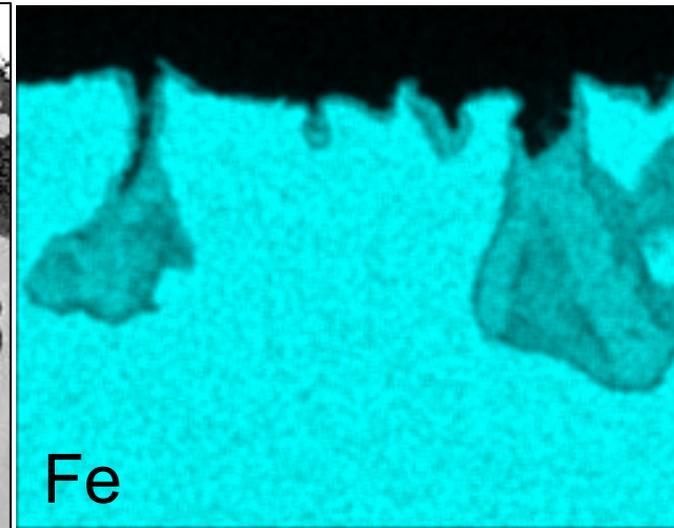
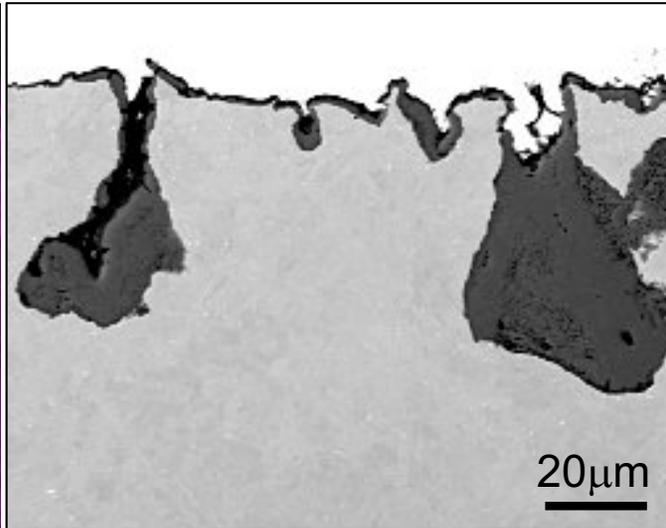
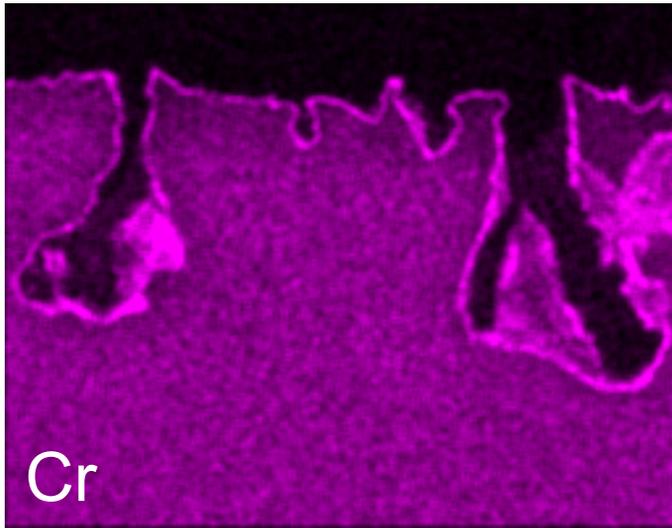
Oxide scale at specimen surface

# Presence of Fine Precipitates in the Affected Zone. Internal Oxidation?



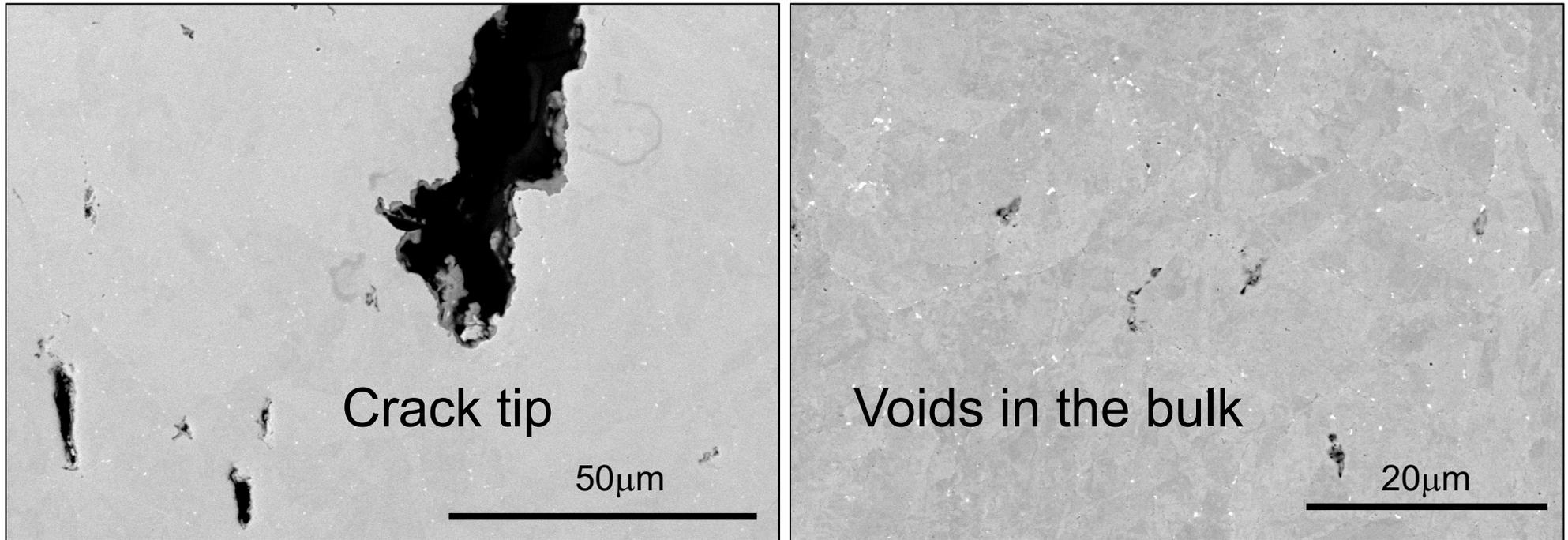
- Need TEM characterization to identify precipitates

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



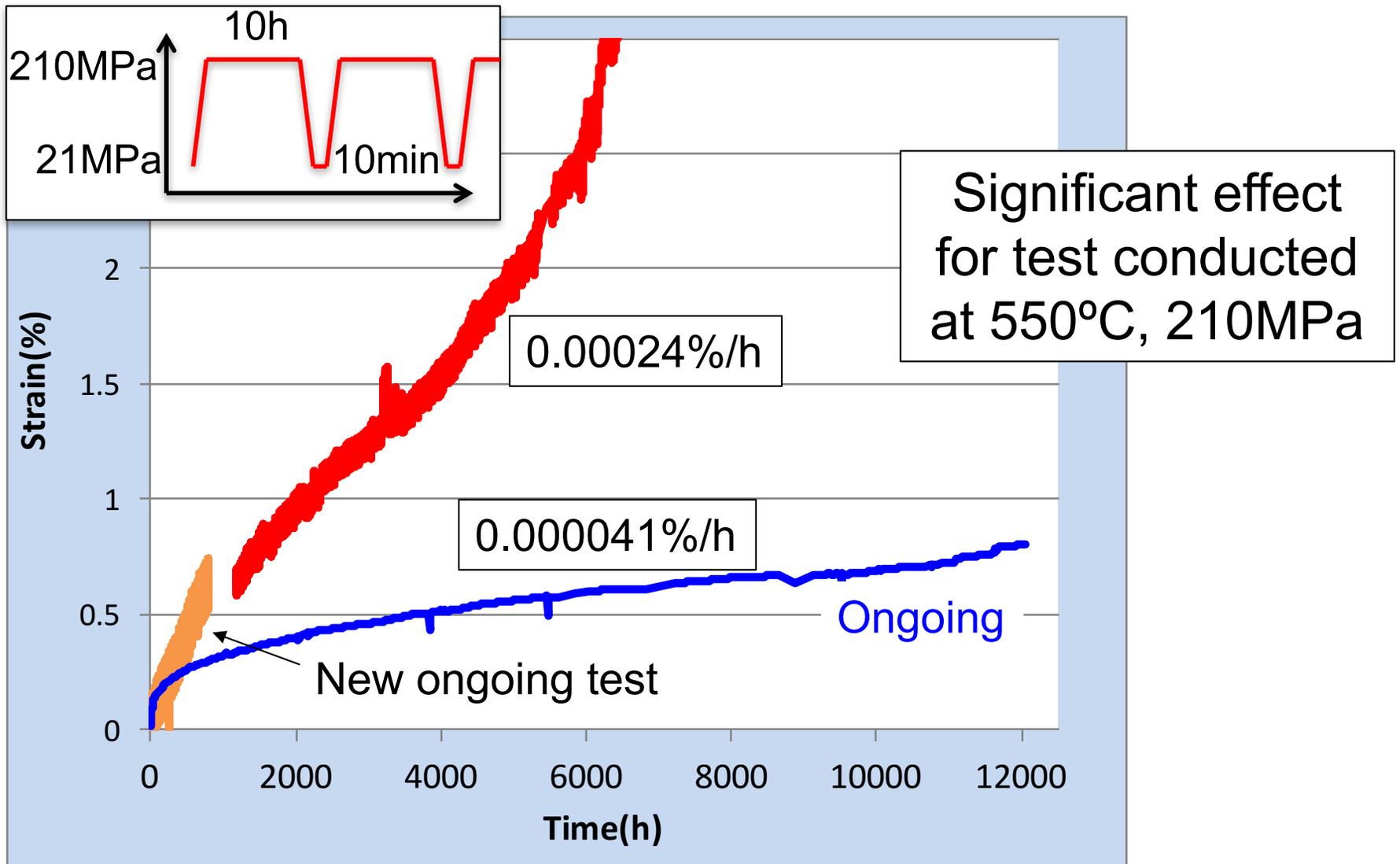
Mostly thin Cr-rich  
oxide layer + Fe  
oxide in oxidized  
cracks

# Interaction Between Crack Propagation & Creep Cavitation for $\pm 0.5\%$ , 10h Hold

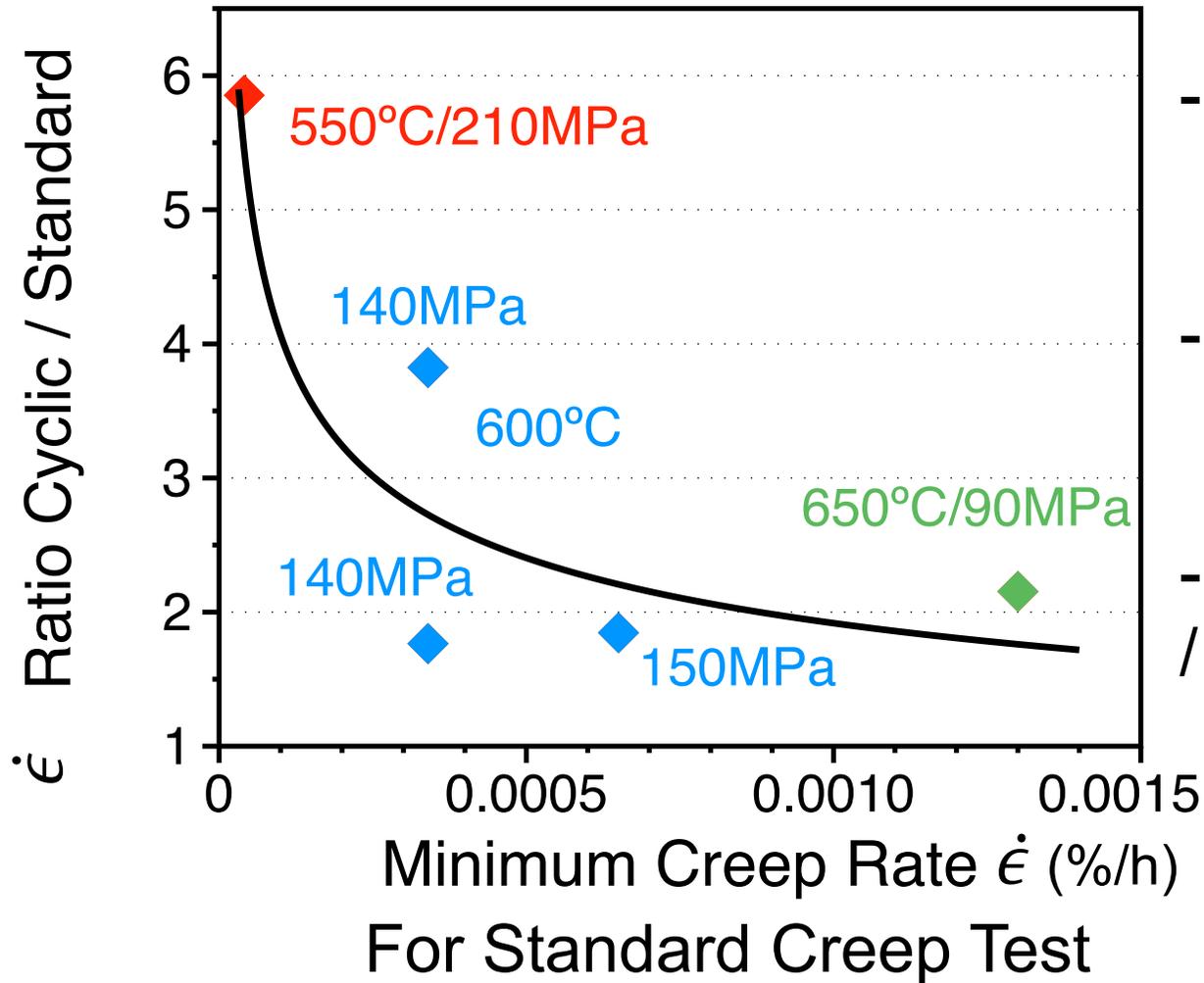


- Lower cycle to failure for 10h hold time test is likely due to
- Fast crack initiation due to oxide scale cracking
  - Effect of creep cavitation on crack propagation

# Effect of Load Cycling on Gr.91 Creep Properties

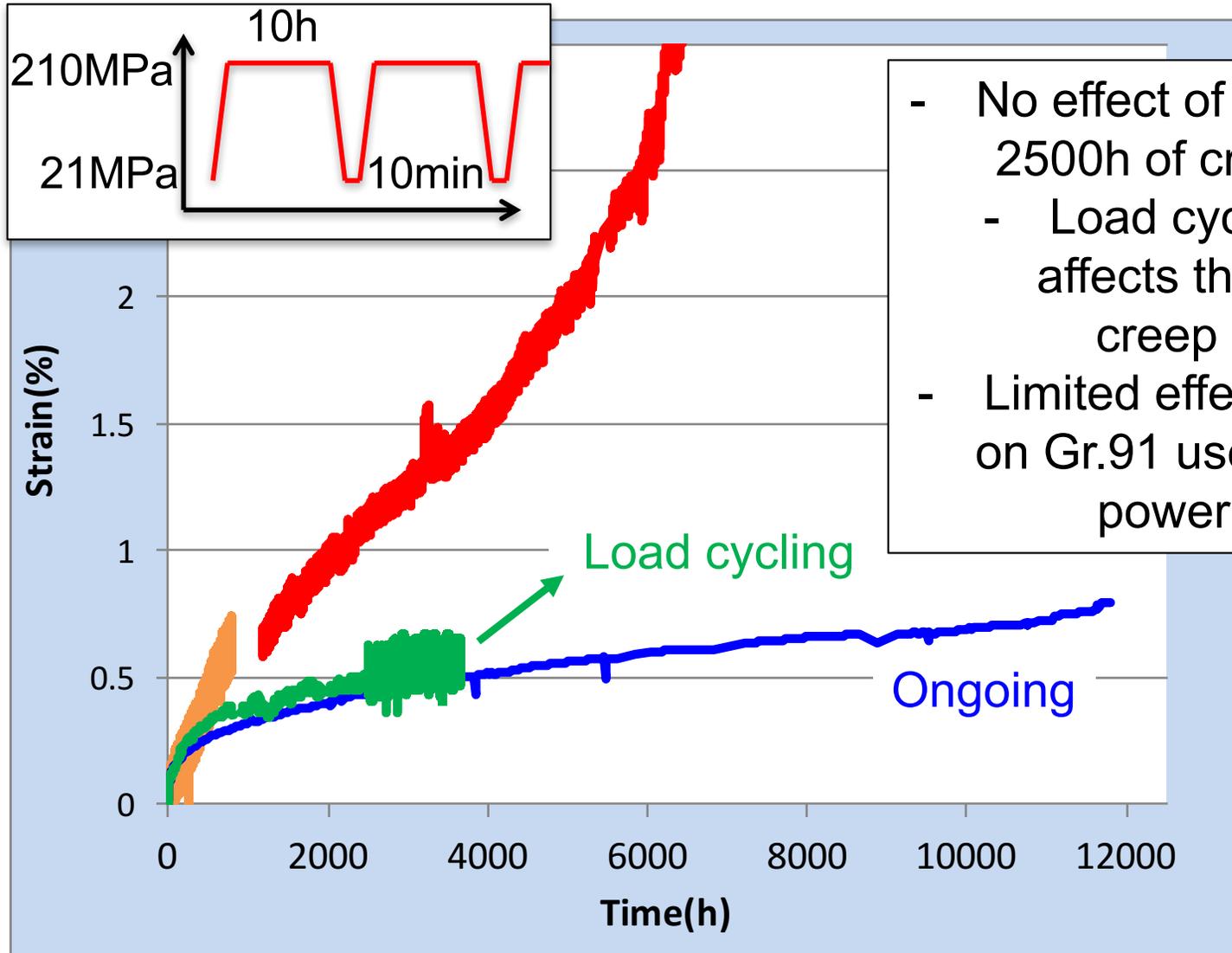


# Significant Increase of Creep Rate for Longer Creep Tests



- Systematic increase ( $\sim X2$ ) of creep rate due to cycling
- Significant variation from one test to another
- Focus on long term test / low creep rate

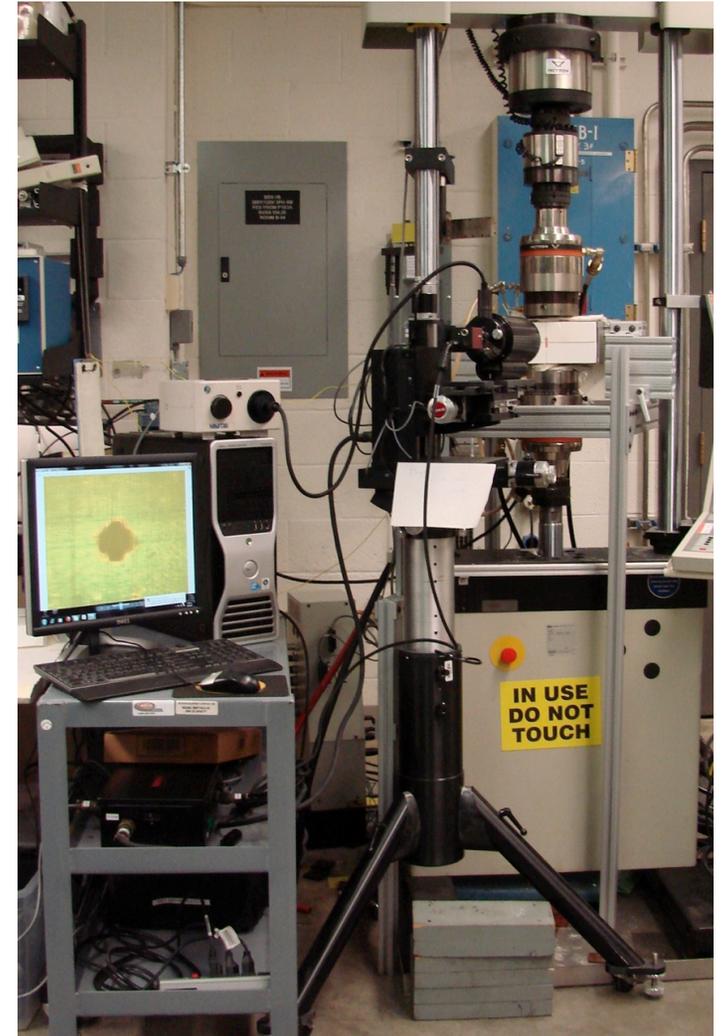
# New Test With Load Cycling Initiated After ~2500h of Creep Testing



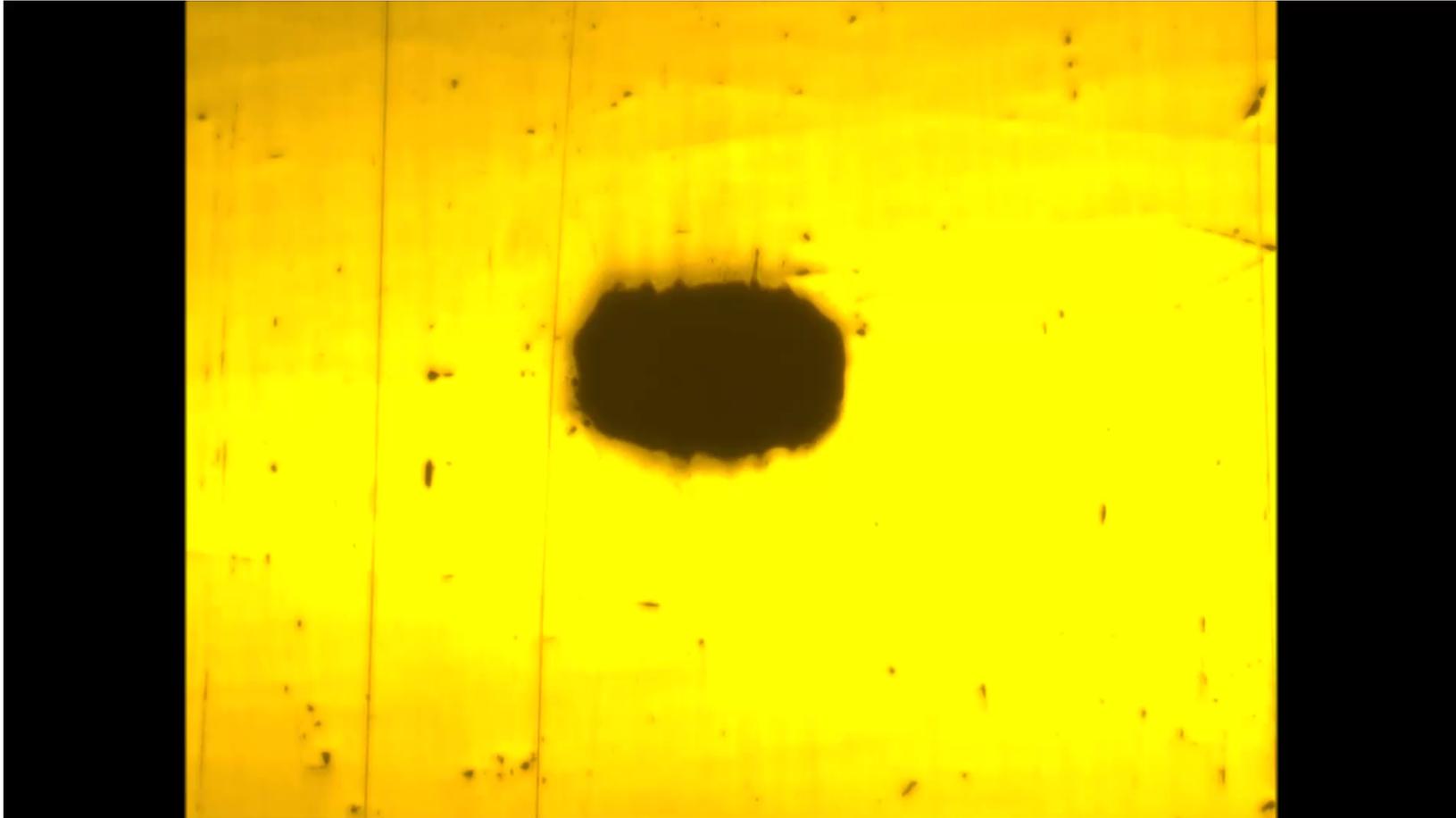
- No effect of cycling after 2500h of creep testing
- Load cycling only affects the primary creep stage?
- Limited effect of cycling on Gr.91 used in current power plant?

# 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
- Crack initiation at room temperature
- High cyclic Fatigue & Creep Fatigue Testing
- In Situ imaging of crack propagation
- Tests conducted at Room and 550°C

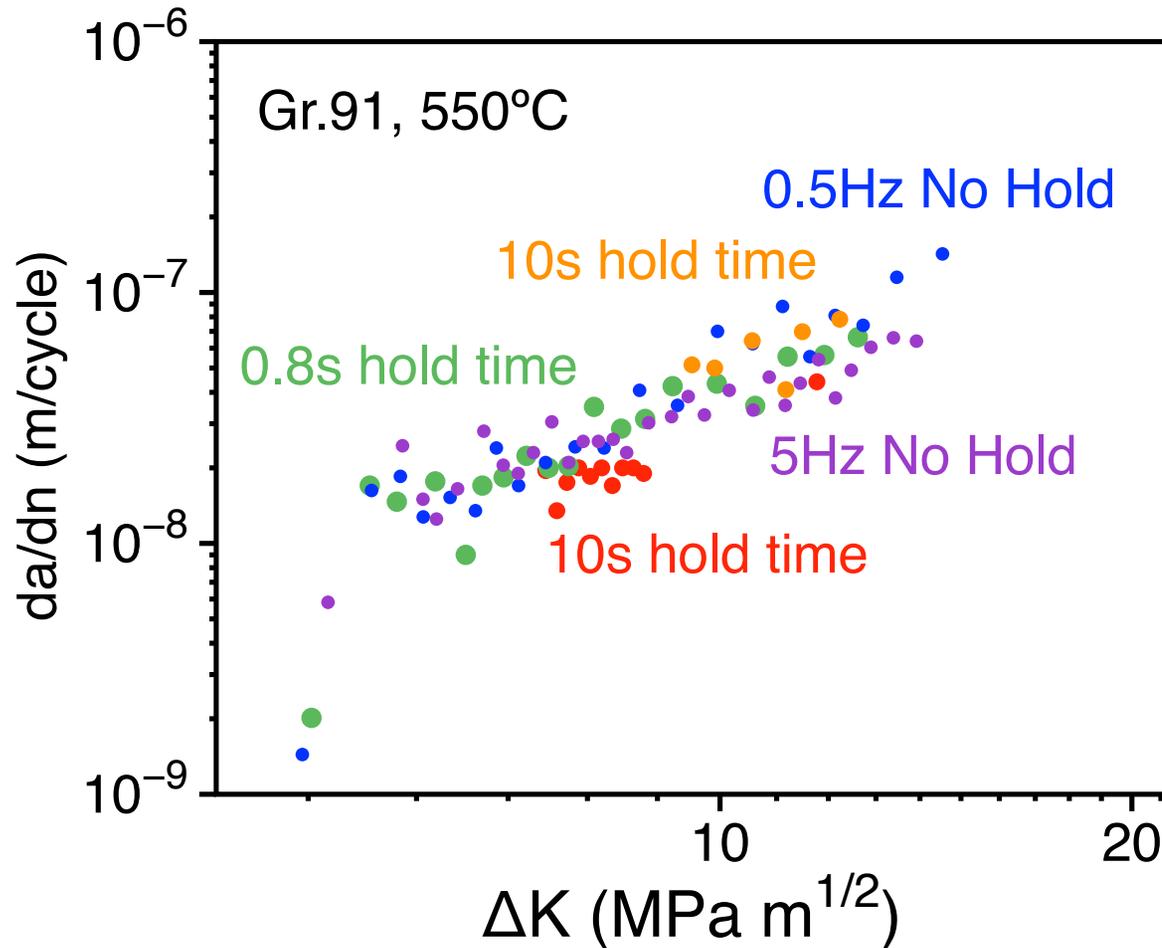


# Crack Growth Imaging at Room T°C



300μm

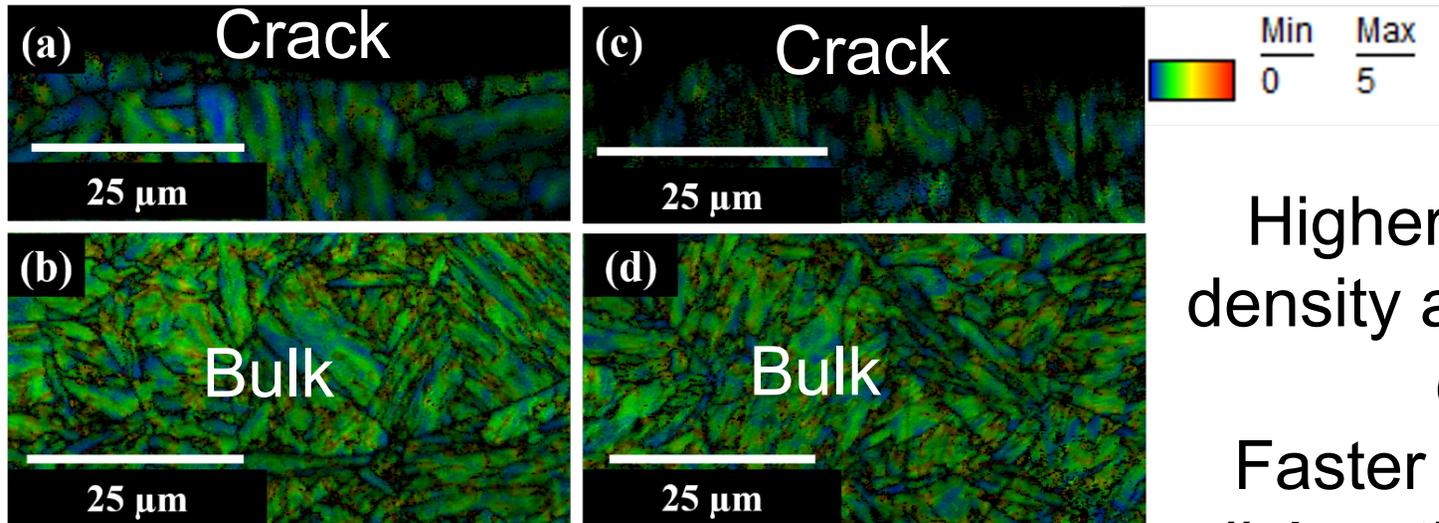
# No Effect of Frequency & Hold Time on Small Crack Propagation



No crack propagation with 100s or 10min hold time

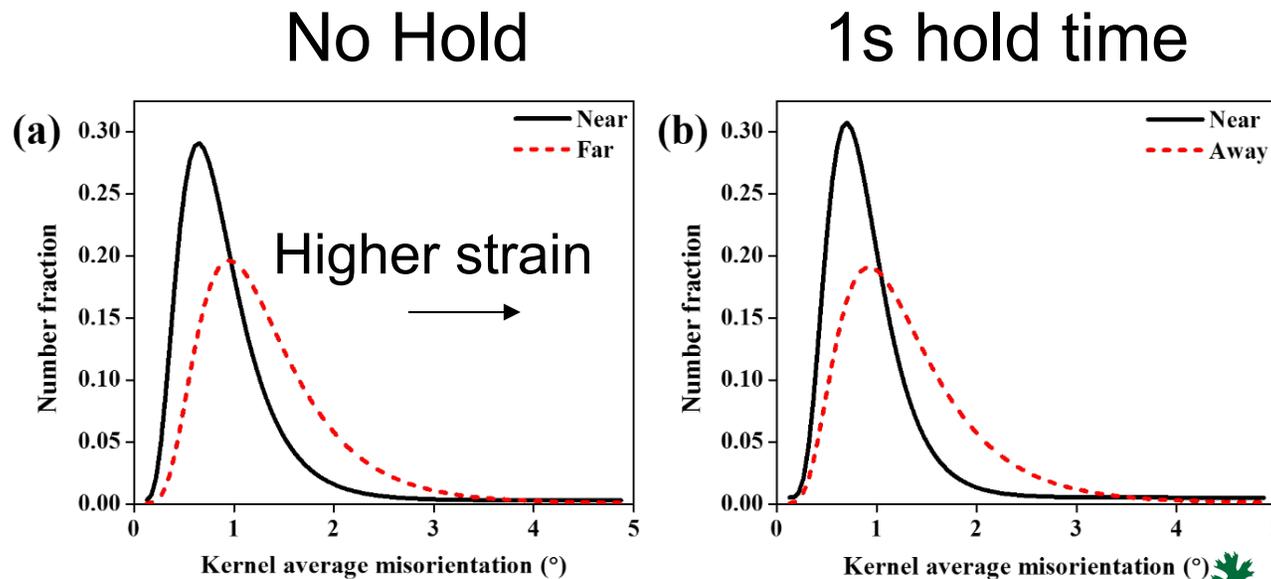
Effect of oxidation or decrease of dislocation density at crack tip?

# EBSD Measurements to assess strain/dislocation density

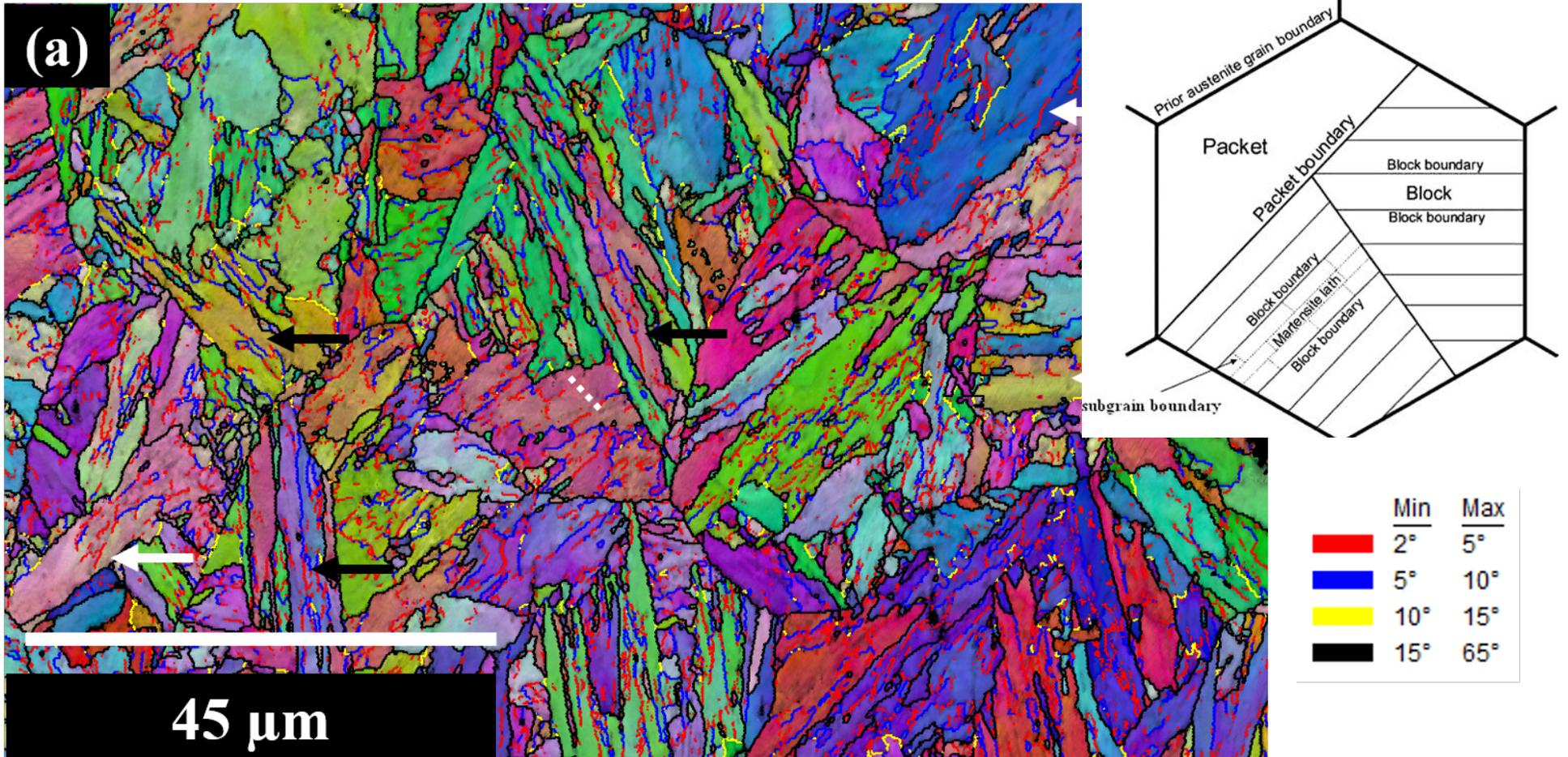


Higher dislocation density away from the crack

Faster decrease of dislocation density at crack tip for creep-fatigue specimens?  
Depends on Gr91 ductility

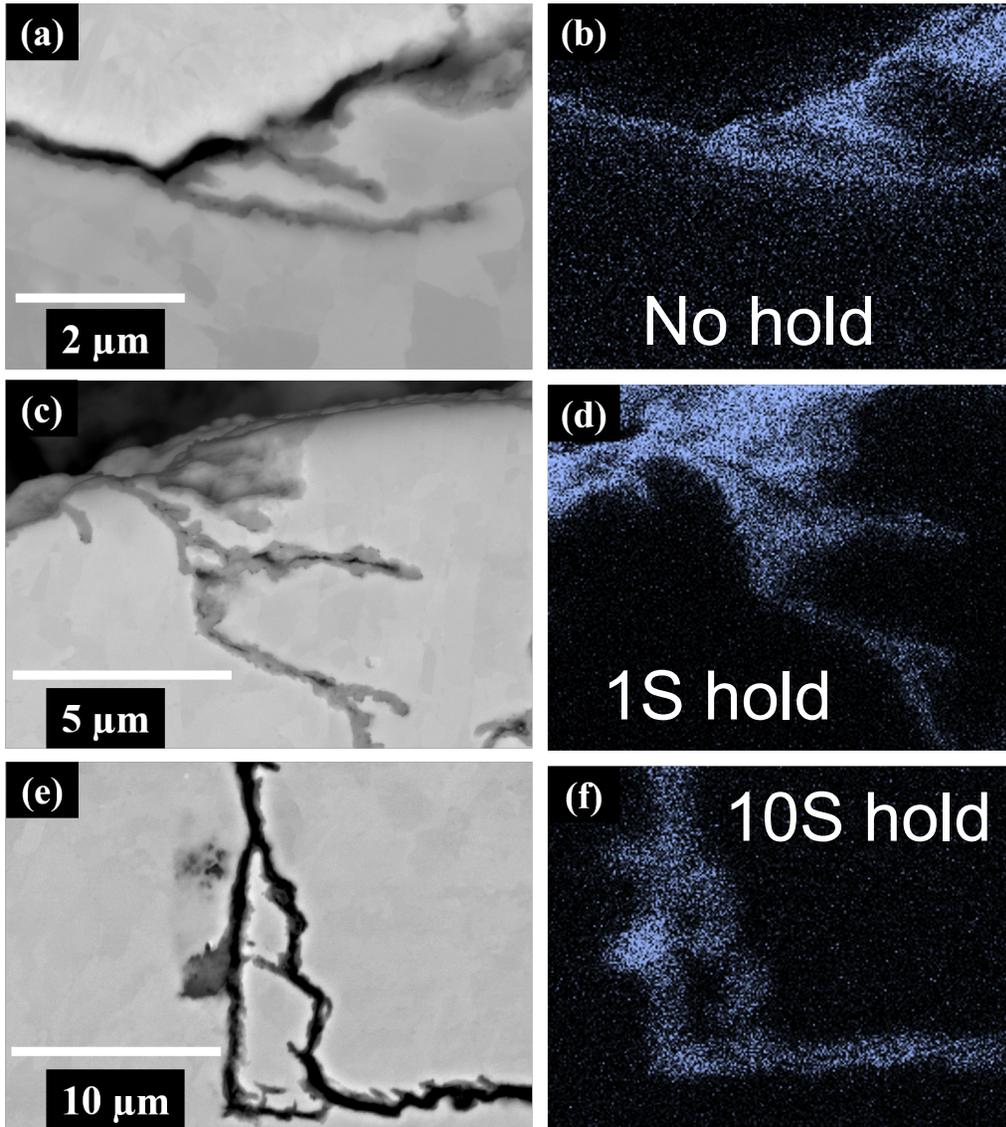


# Extensive Characterization of Boundaries Misorientation



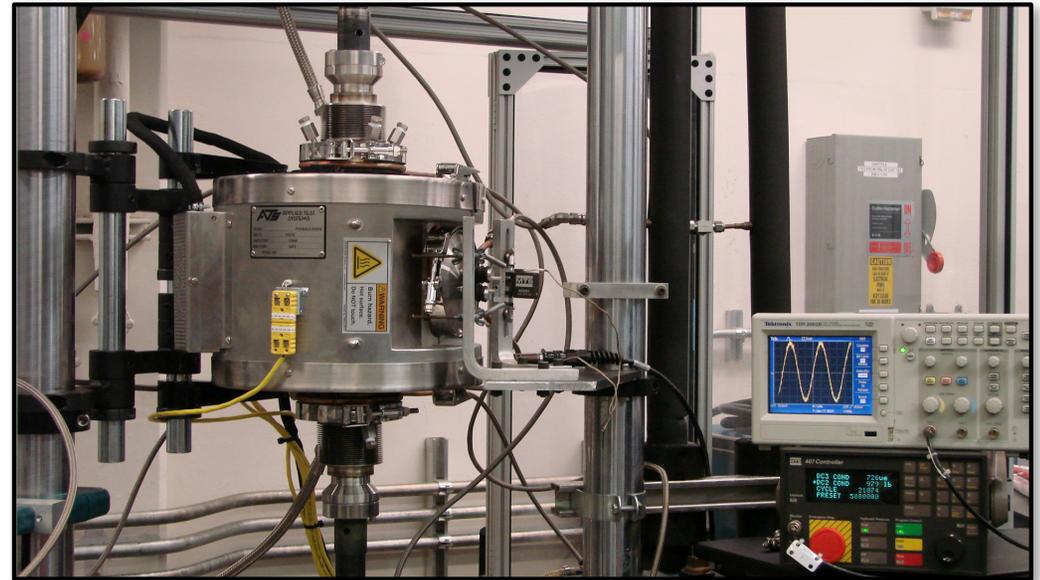
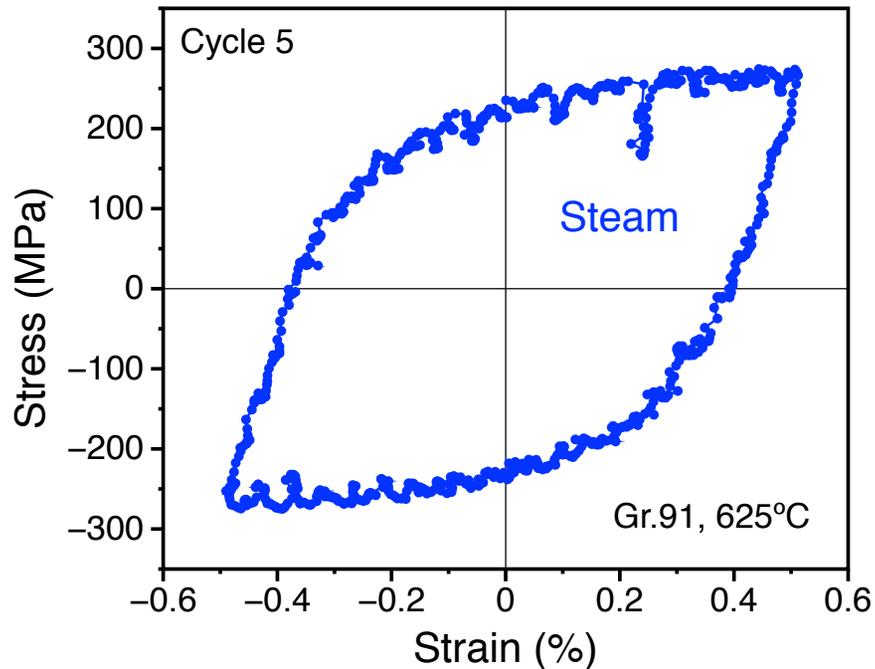
Misorientation between the sub-grains boundaries or block boundaries increased due to hold time

# Significant Oxidation at the Crack Tip During Testing



Higher dislocation density  
away from the crack  
Faster decrease of  
dislocation density at  
crack tip due to hold  
time?

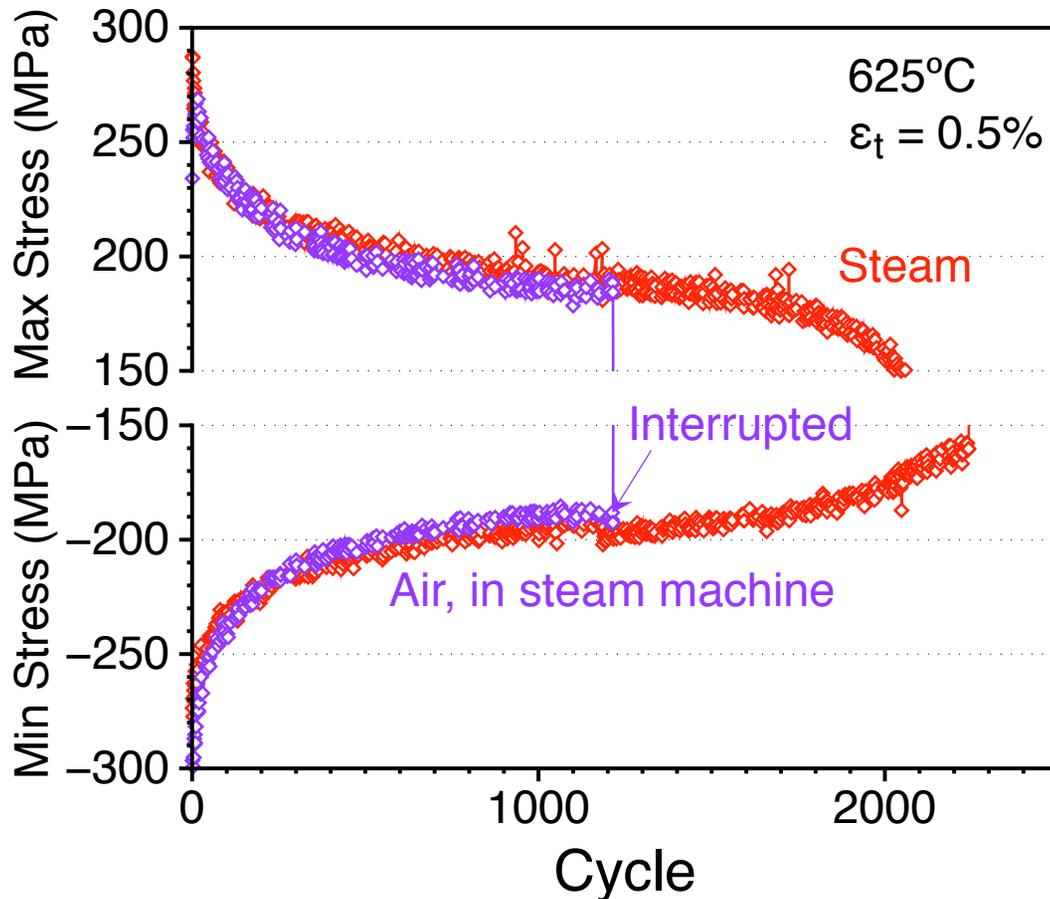
# New test facility to evaluate LCF resistance in aggressive environments



Stress-strain curve, 9Cr-1Mo ferritic steel, 625°C, fully reversed 1% total deformation, Steam

Sealed chamber inside furnace and extensometer to provide feedback signal for system operation under strain control

# Similar Min-Max Curves In Air and Steam, 625°C, 0.5% Strain, No Hold



- But issue with specimen buckling and signal stability
- Machine alignment is ongoing

# Conclusion

- Significant effect of hold time on Gr.91 fatigue lifetime
  - Fast crack initiation due to oxide formation and cracking
  - Interaction between Cavity formation & crack propagation
- Will improve creep-fatigue lifetime modeling based on experimental data in collaboration with 3FEAA118 (Xinghua)
- Long term cyclic creep affects Gr.91 primary creep stage
- Longer hold time lead to small crack propagation arrest
- New rig to conduct LCF testing in corrosive environments
- Future work: Focusing on key industry needs