Effects of Microstructure Evolution on Deformation, Fracture, and Creep-Fatigue Life of a 25Ni-20Cr Austenitic Stainless Steel (Alloy 709)

Ty Porter, PhD, PE

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Henry O. Fuchs Student Award

SAE FD&E Spring Meeting

Ty Porter, PhD, PE

- Education
  - BS in Mechanical Engineering at CSM, 2012
  - PhD in Metallurgical and Materials Engineering at CSM, 2019
- Previous Industry Experience
  - Precision Castparts Corp. (2012-2013)
  - ESCO Corp. (2013-2015)
- Exponent, Inc (2019-present)
  - Senior Engineer in Materials and Corrosion Practice
  - Consultant in failure analysis and materials engineering solutions in a wide range of industries
  - Denver, CO office

Mentors, Collaborators, and Funding

- *Advisor*: Prof. Kip Findley (CSM)
- *Committee*: Profs. Michael Kaufman (CSM), Kester Clarke (CSM), Aaron Stebner (GIT), and Dr. Richard Wright (INL)
- *Collaborators*: Drs. Zhiyang Wang (ANSTO) and Mike McMurtrey (INL)
- *Funding*:
    - Advanced Steel Processing and Products Research Center (ASPPRC)
    - US DOE Nuclear Energy University Program

CSM *ASPPRC*

NEUP Nuclear Energy University Program
U.S. Department of Energy

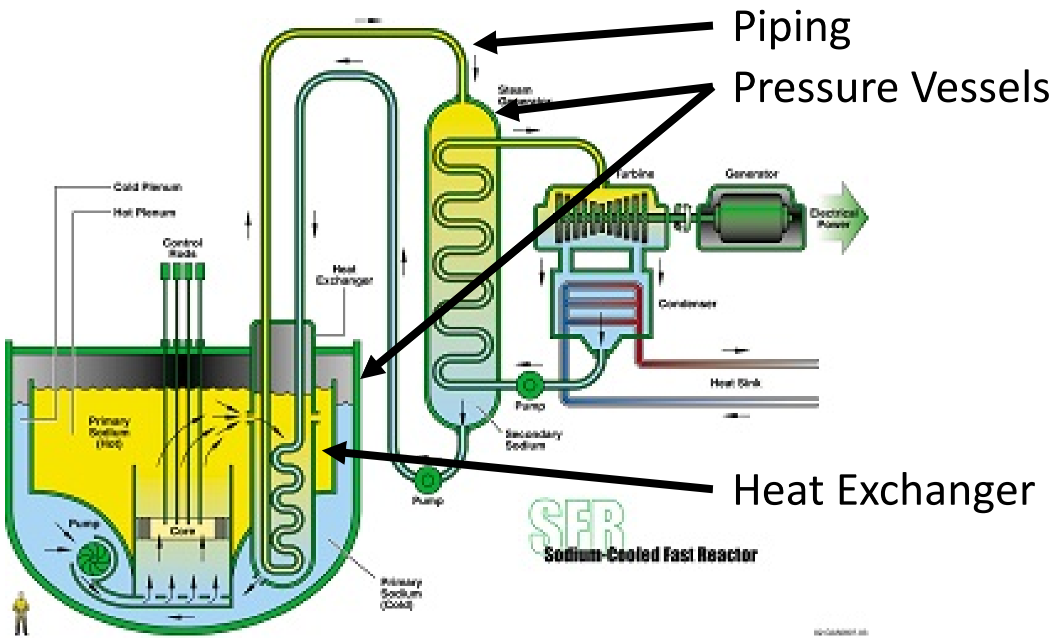
Motivation

The interaction of load, time, and environment along with materials selection, geometry, processing, and residual stresses creates a wide range of synergistic complexity and possible failure modes in all fields of engineering.

Metal Fatigue in Engineering, 2nd Ed.
*R. Stephens, A. Fatemi, R. Stephens, and **Henry O. Fuchs***

Project Relevance and Background

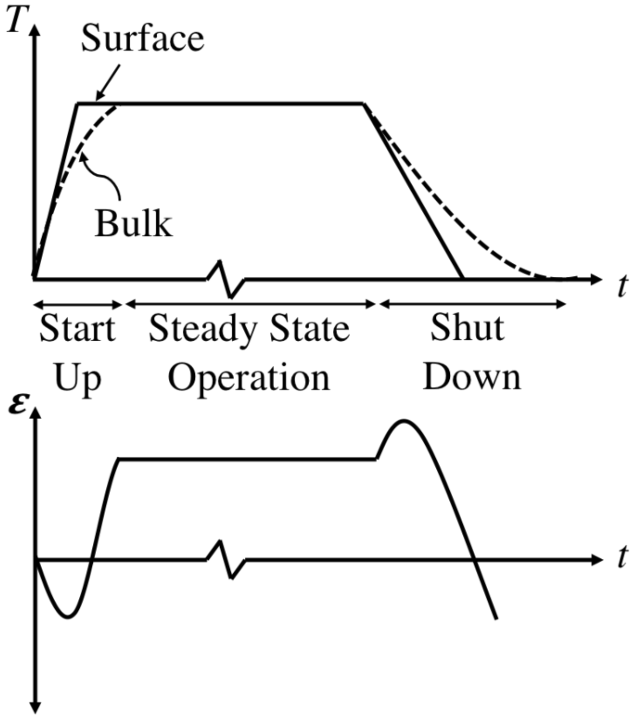
- Several candidate next generation (Gen IV) nuclear reactor designs
- Sodium cooled fast spectrum reactors (FSRs) operate at 550 °C and have a desired life of 50 years
- Structural components are made from highly corrosion-resistant and creep-resistant alloys



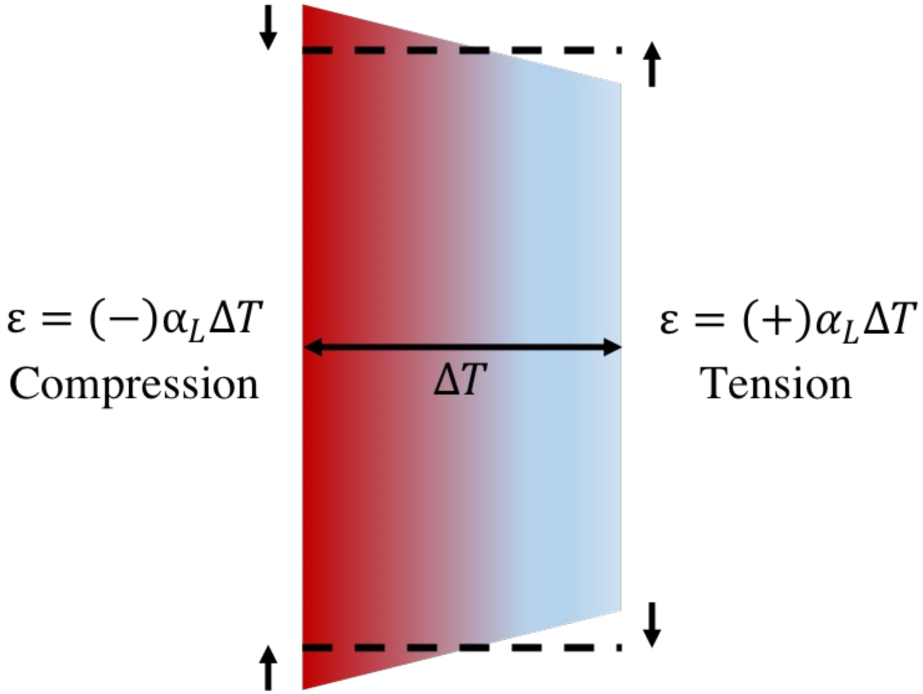
Generation IV International Forum, 2017

Creep-Fatigue Review

- Thermal low cycle fatigue (LCF) occurs during transient start-up and shut-down operations in thick section structural components
- Creep conditions exist during steady-state operation
- Austenitic stainless steels are particularly sensitive to LCF

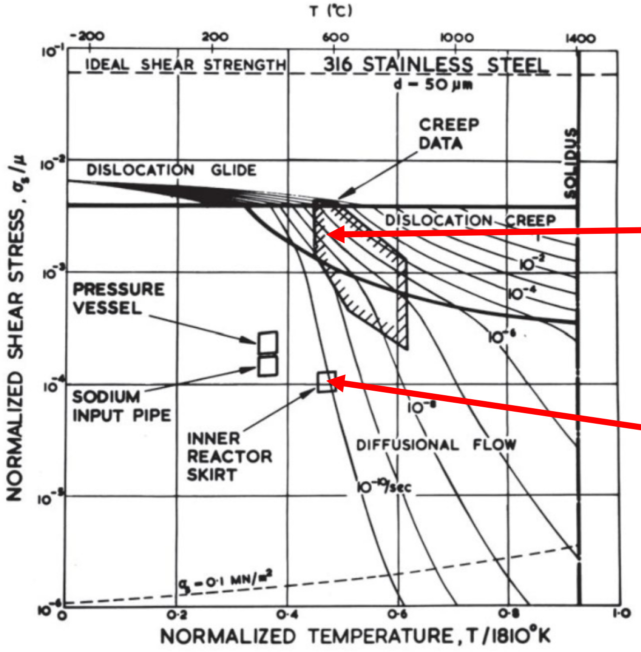


Adapted from P. Rodriguez *et al.* 1993

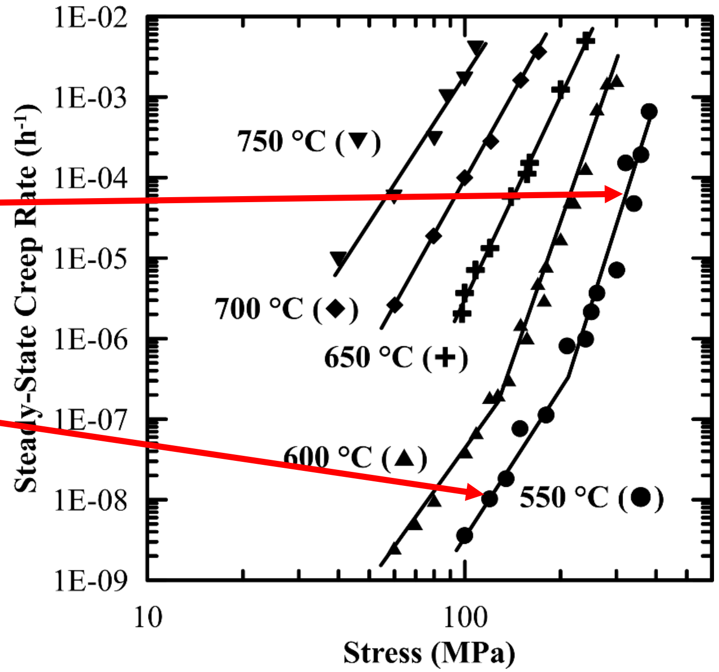


Accelerated Testing

- Long-term life prediction methods for design are based on extrapolation of laboratory data (short term tests)
- Laboratory creep and creep-fatigue tests are conducted under conditions of accelerated damage accumulation (high temperature, stress, strain, strain rate, etc.)
- Mechanisms of deformation and damage may not be the same under accelerated conditions compared to service conditions



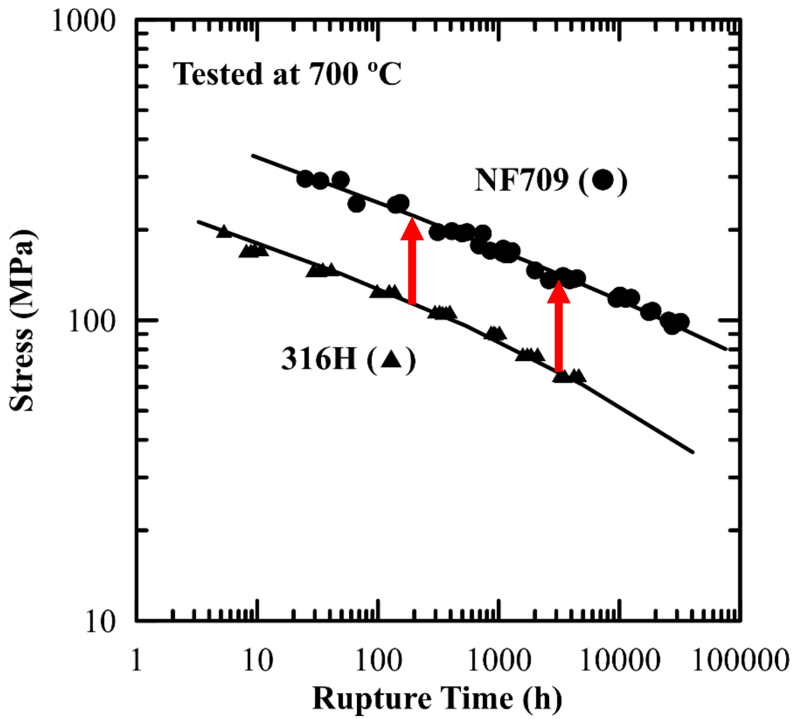
Frost *et al.* 1977



Rieth *et al.* 2004

Alloy 709 Background

- Alloy 709 (20Cr-25Ni-1.5Mo-0.25Nb-0.15N) is a solid solution and precipitation strengthened austenitic stainless steel with excellent high temperature creep strength
- Alloy 709 has significantly higher creep resistance than 316H
- Candidate structural alloy for Gen IV FSRs
- Limited creep-fatigue research



Yamamoto *et al.* 2012

~2x creep strength over Type 316H at 700°C

Research Objective and Methods

Objectives:

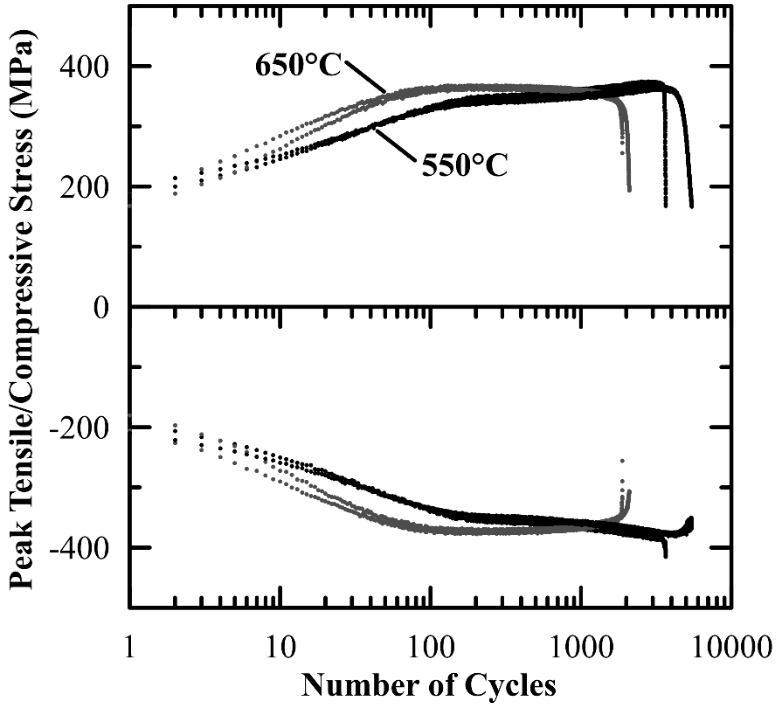
- Develop an understanding of the cyclic deformation and damage mechanisms at the relevant service temperature (550 °C) and at an accelerated test temperature (650 °C)
- Investigate the influence of microstructural evolution on creep-fatigue performance

Methods:

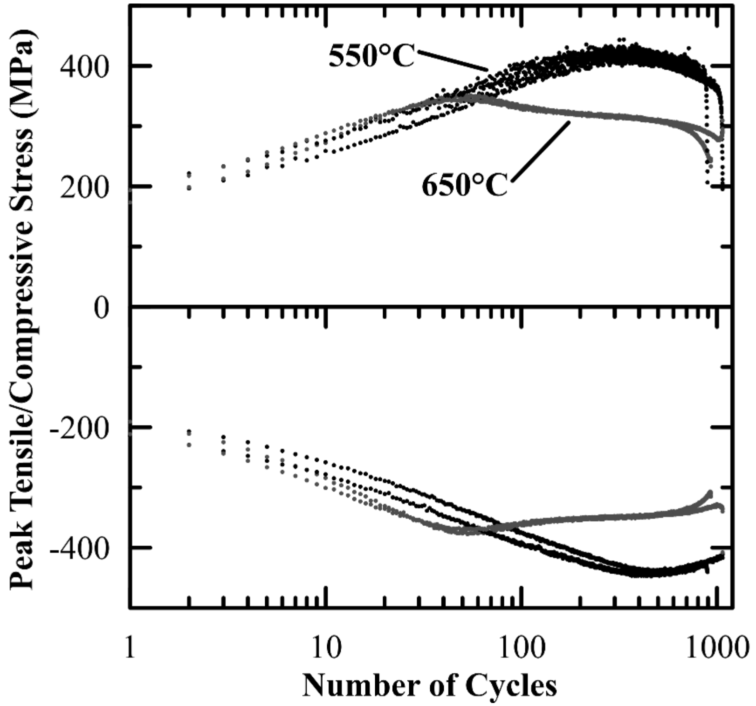
- LCF and creep-fatigue (30 min hold) tests at 550 and 650°C with **solution annealed (SA)** microstructure
- Same test conditions with a **thermally aged** microstructure

Initial LCF and Creep-Fatigue Test Results

$\Delta\varepsilon$=1%, $\dot{\varepsilon}$=10^{-3}, **LCF**



$\Delta\varepsilon$=1%, $\dot{\varepsilon}$=10^{-3}, **Creep-Fatigue**



Test Condition		**Average Cycles to Failure, N_f (cycles)**
LCF	550°C, no hold	4296
	650°C, no hold	1965
Creep-Fatigue	550°C, 30 min hold	975
	650°C, 30 min hold	1000

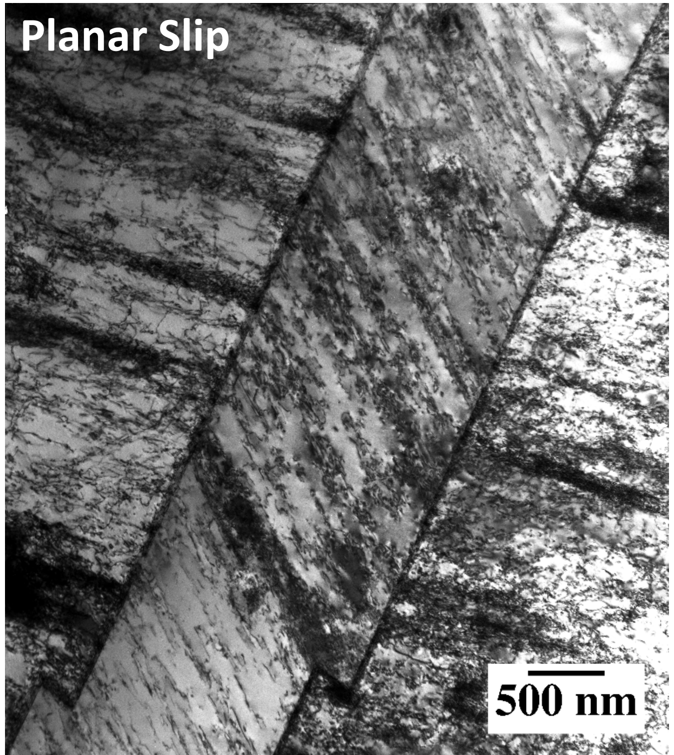
~4x life reduction at 550°C

~2x life reduction at 650°C

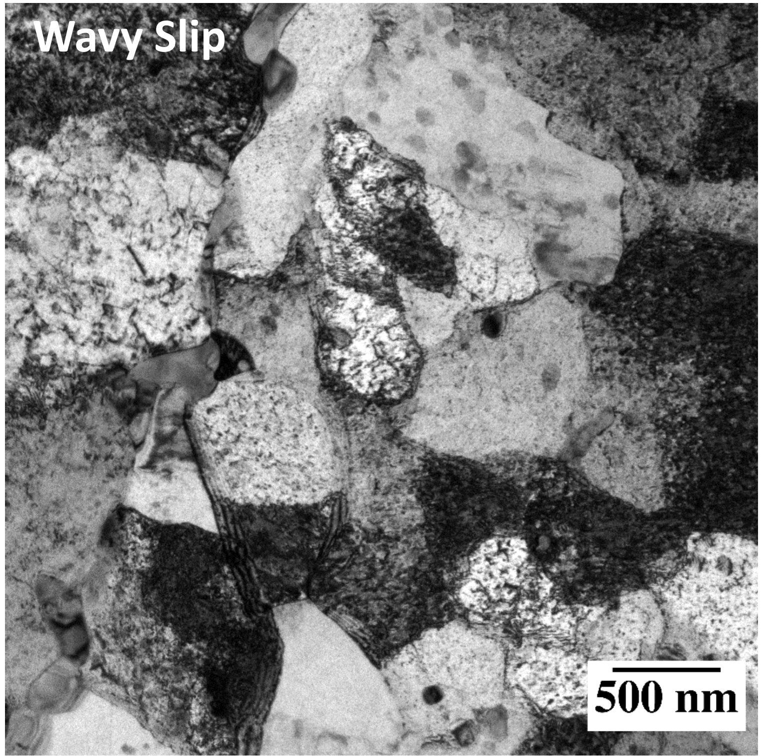
Deformation and Damage

- **At 550°C**: Primarily intergranular fracture, **At 650°C**: mixed transgranular/intergranular fracture
- Approximately twice the grain boundary damage (GB cracking) at 550°C compared to 650°C
  - *Less accumulated creep strain at 550 °C, but higher tensile stresses and no cyclic softening*
- Microstructures at end of life show significantly different deformation structures, slip behavior

BF TEM: 550°C Creep-Fatigue



BF TEM: 650°C Creep-Fatigue

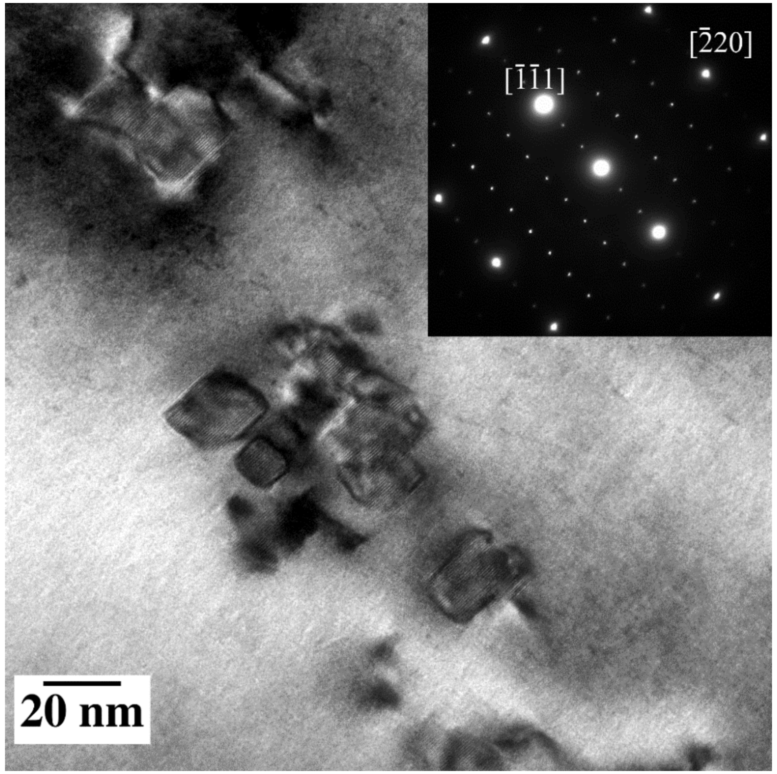


Porter *et al.*, 2019

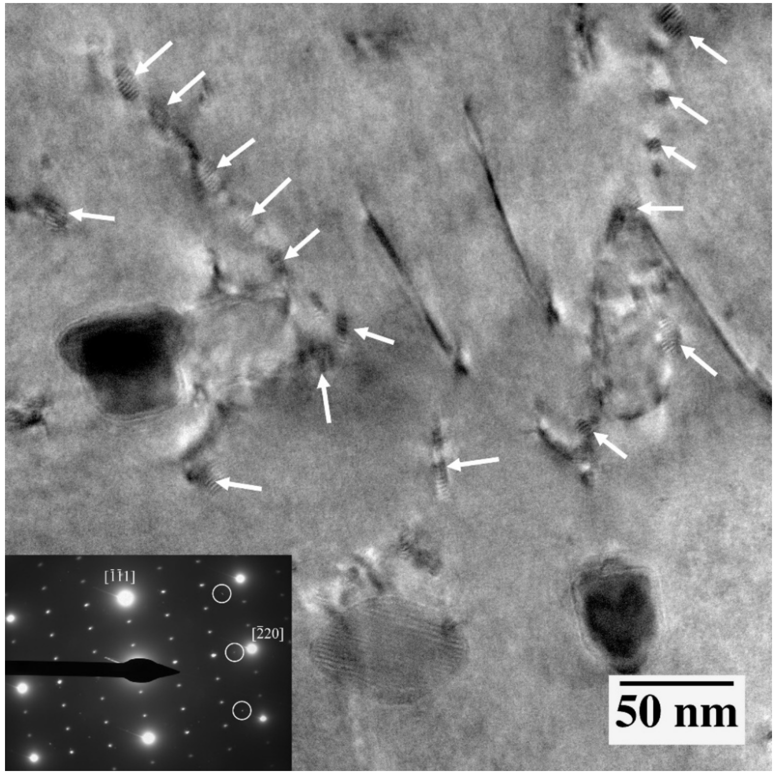
Microstructural Evolution

- Significant differences in precipitation at end of fatigue and creep-fatigue tests between 550 and 650°C

TEM BF: **550°C** Creep-Fatigue
$M_{23}C_6$ ~ 10-20 nm; $\boldsymbol{f_v}$ ***= 0.25%***
No M(C,N) observed



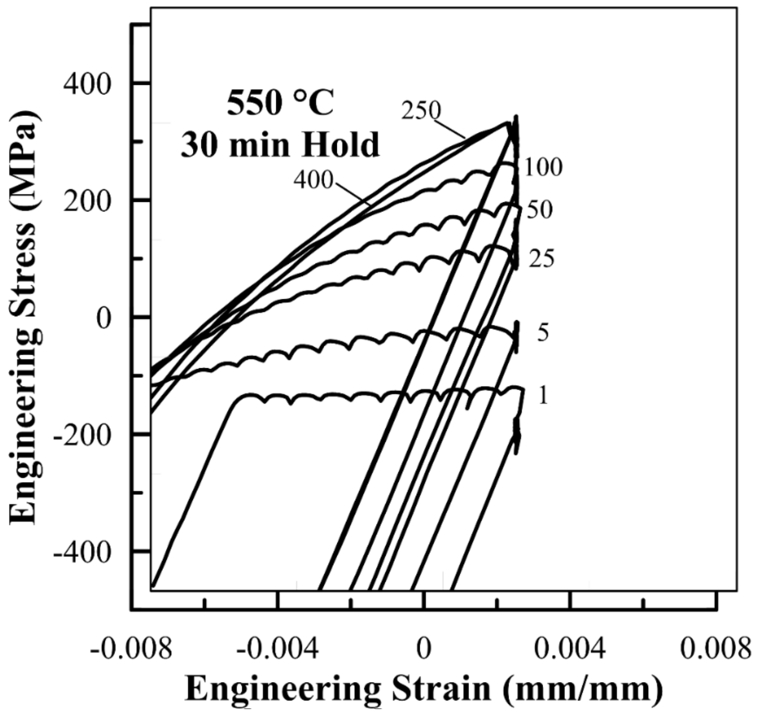
TEM BF: **650°C** Creep-Fatigue
$M_{23}C_6$ ~ 50 nm; $\boldsymbol{f_v}$ ***= 0.80%***
M(C,N) ~ 5-10 nm

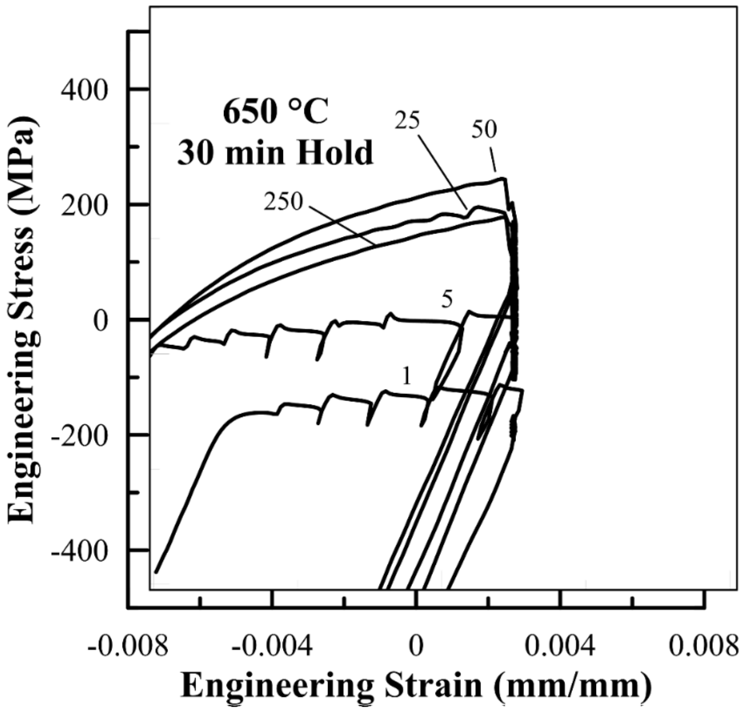


Porter *et al.*, 2021

Serrated Flow and DSA

- Serrated flow in hysteresis loops → indicative of dynamic strain aging (DSA)
- Common in austenitic steels with high interstitial concentration
  - i.e. 316L(N)
- Serrations disappear after some number of cycles, but differs at different temperatures

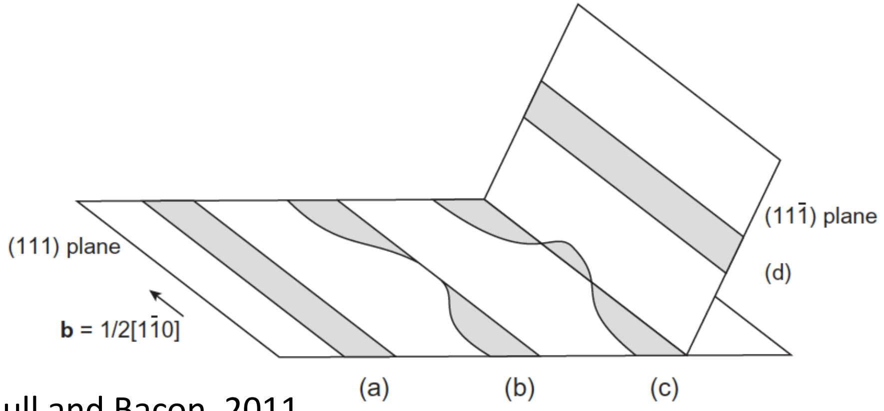




Effect of Solute on Deformation in FCC Metals

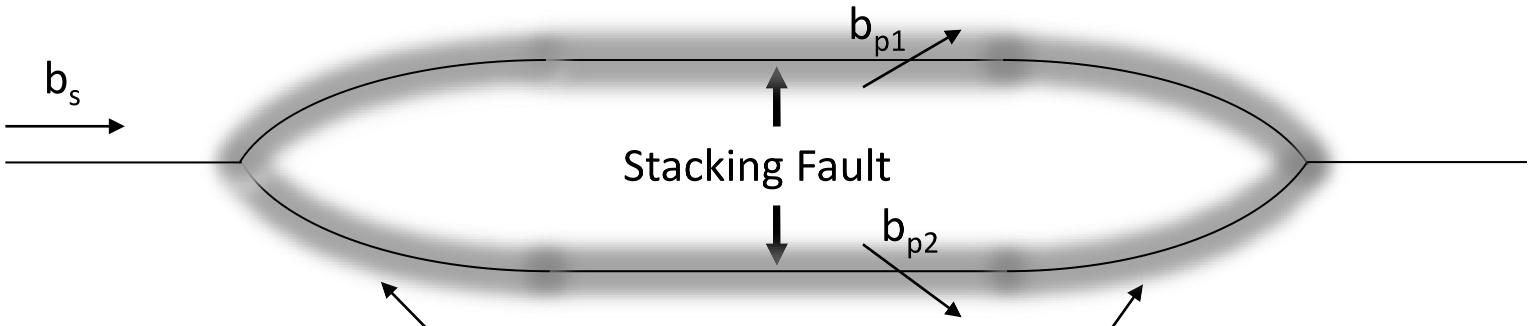
- Cross-slip is an important mechanism for dynamic recovery and subgrain formation
- For cross-slip in FCC materials, dislocation partials must constrict (SFE is important!)

Dislocation Cross-Slip



Hull and Bacon, 2011

Dissociated Partial Screw Dislocation



Total force for partial dislocation constriction for cross-slip:

$$F_{total} = F_{replusion} - \gamma_{SFE} + \boldsymbol{2F_{friction}}$$

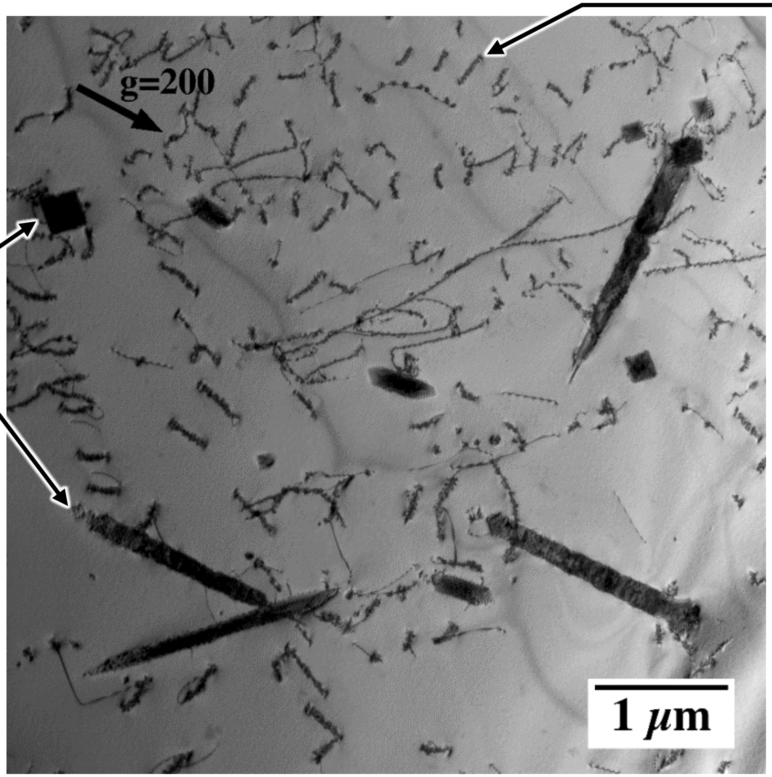
where $\boldsymbol{F_{friction}} = \boldsymbol{f(Solute\ Concentration)}$

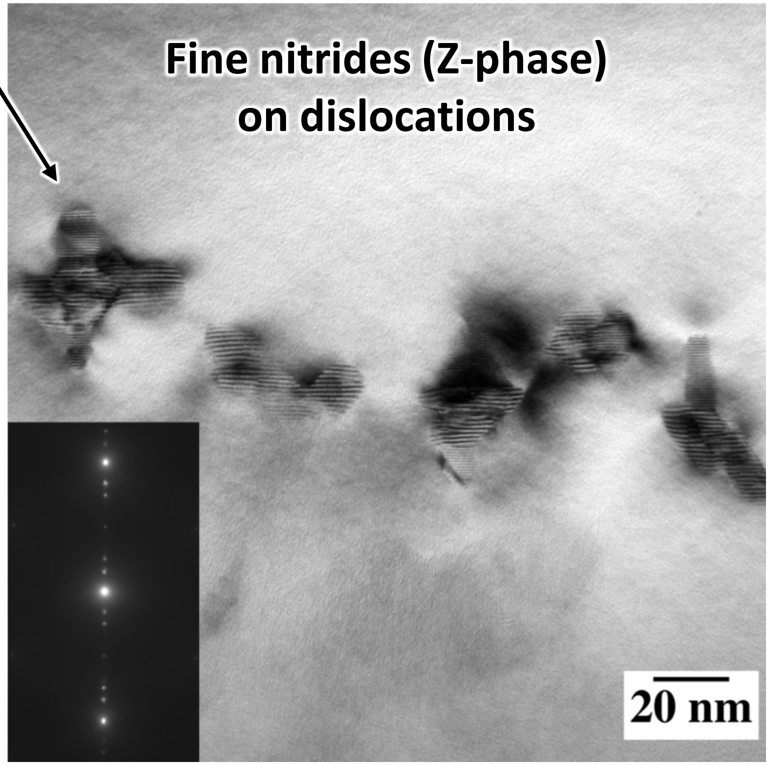
Hong and Laird, 1990

Thermal Aging Treatment

- Effect of dynamic precipitation and solute concentration on creep-fatigue performance and deformation was investigated further by pre-aging the microstructure → ***Can we promote cross-slip at 550 °C?***
- Aged at 750 °C,100 h → Coarse $M_{23}C_6$ (f_v=0.87%) and fine Z-phase (CrNbN)

Coarse carbides ($M_{23}C_6$)

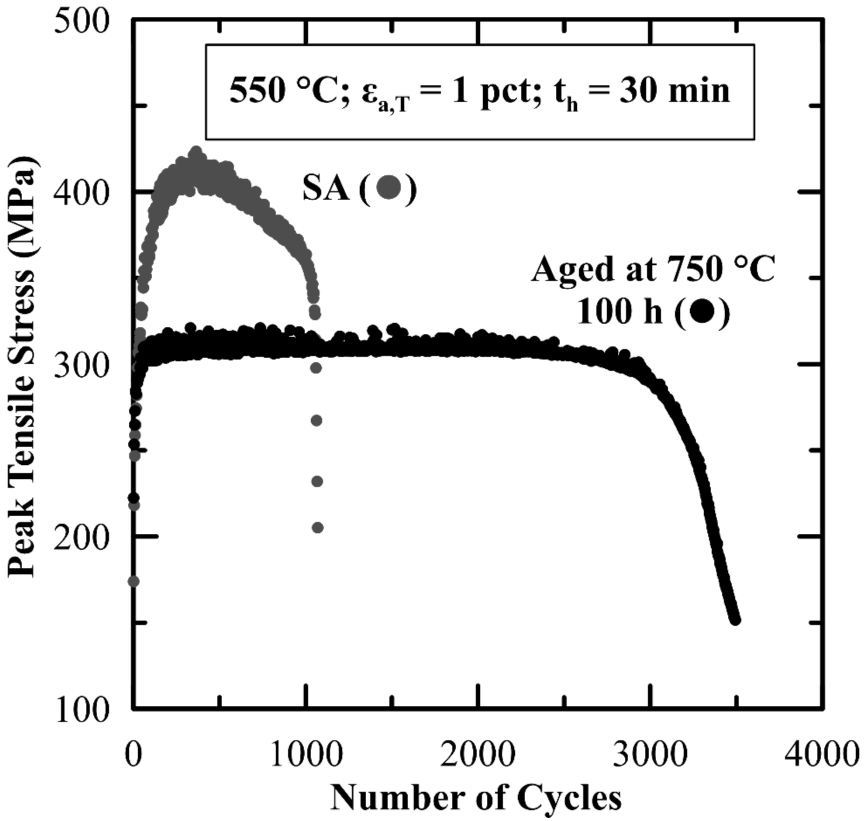


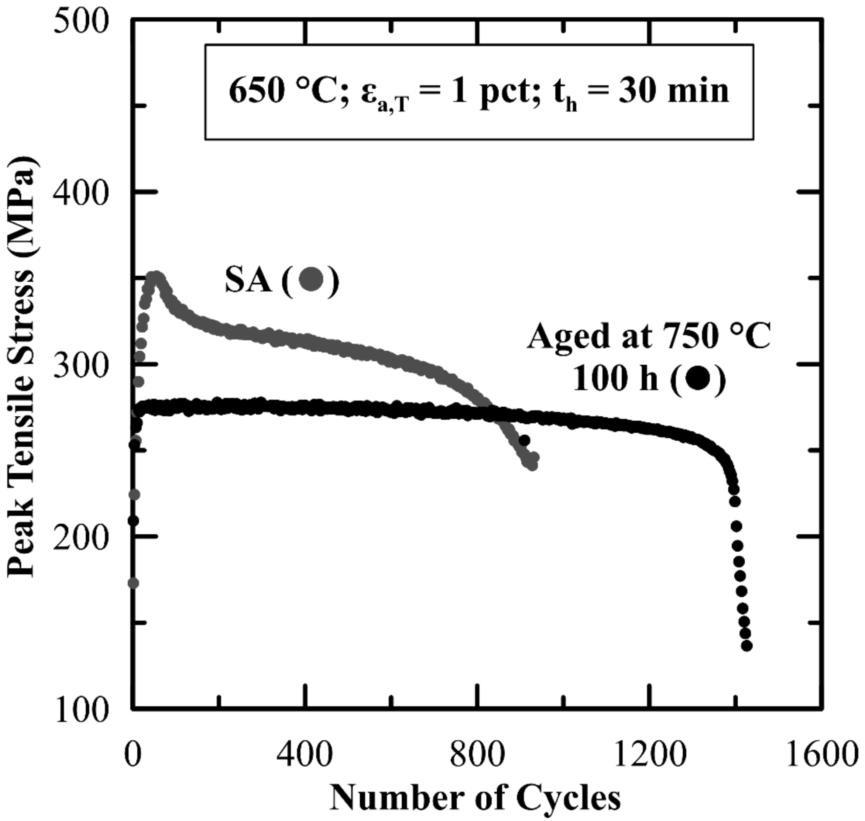


Porter *et al., 2021*

Creep-Fatigue with Aged Material

- Creep-fatigue tests with 30 min holds at 550 and 650 °C with SA and pre-aged microstructure
- Pre-aging results in stable creep-fatigue behavior and significantly longer lives (>3X at 550 °C, ~1.5X at 650 °C)



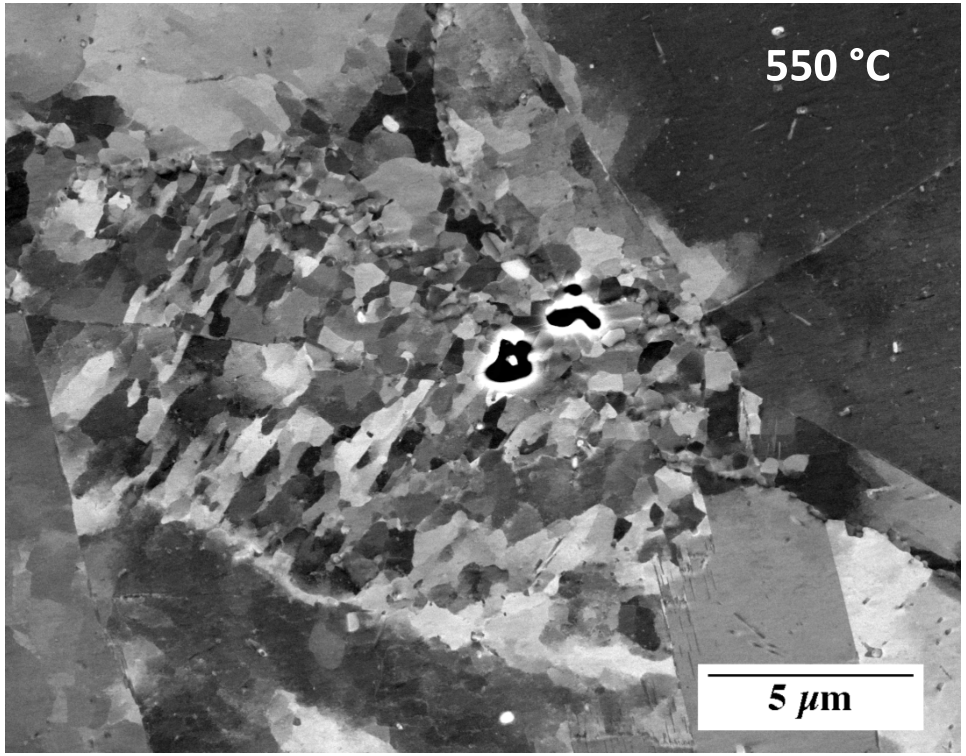


Porter *et al.*, unpublished research

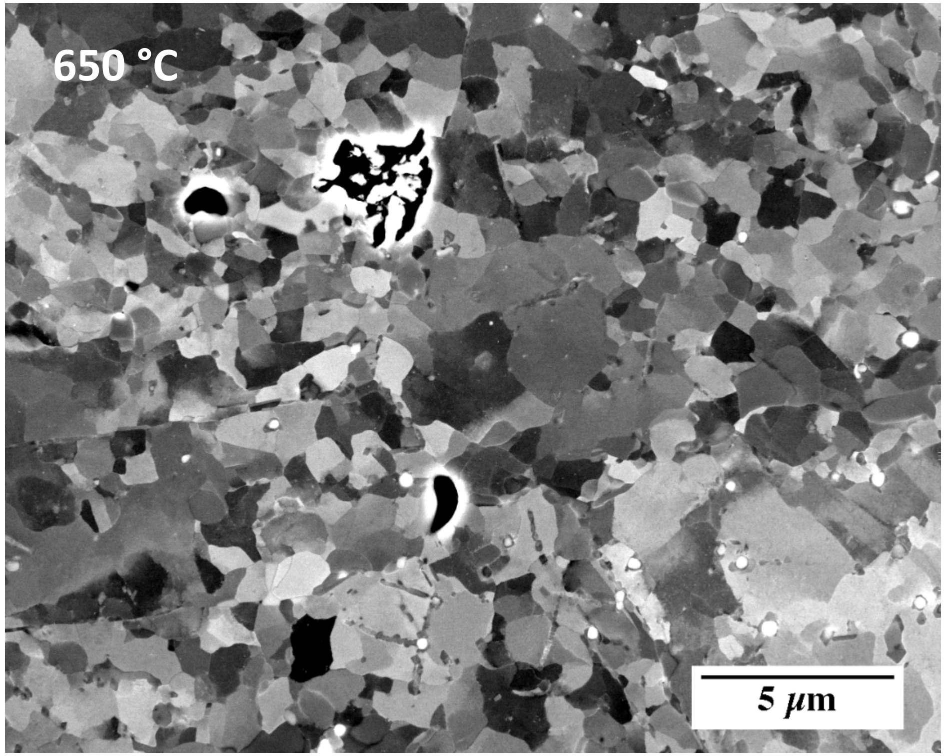
Microstructures at Failure of Aged Material

- Pre-aging results in dynamic recovery (subgrain formation) at 550°C due to increased cross-slip
- Internal damage consists of short blunted cracks or creep voids as a result of increased plastic deformation (dynamic recovery) adjacent to grain boundaries

SEM ECCI: **550°C pre-aged**



SEM ECCI: **650°C pre-aged**

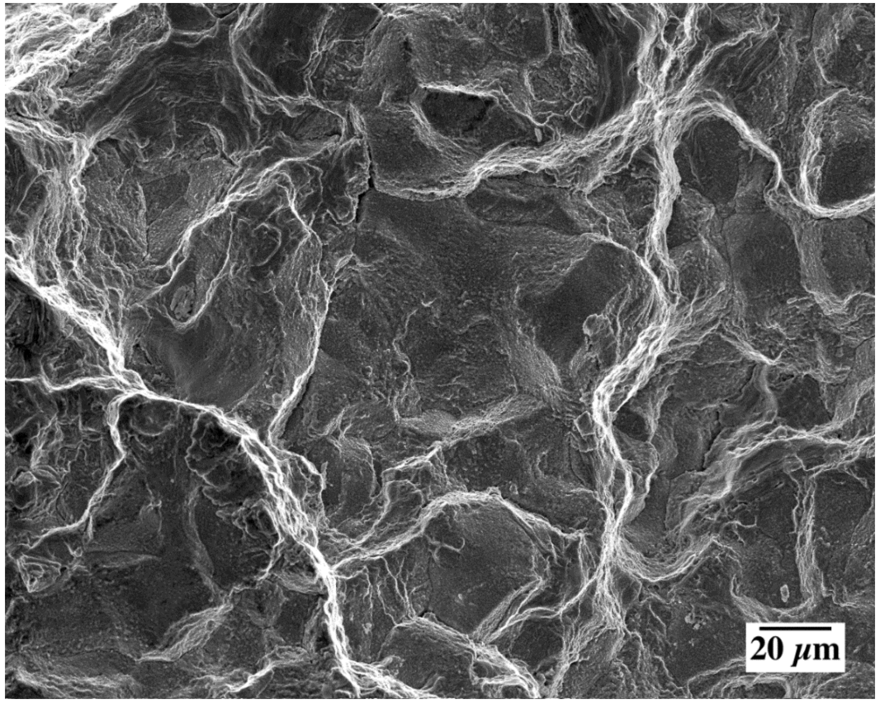


Porter *et al.*, unpublished research

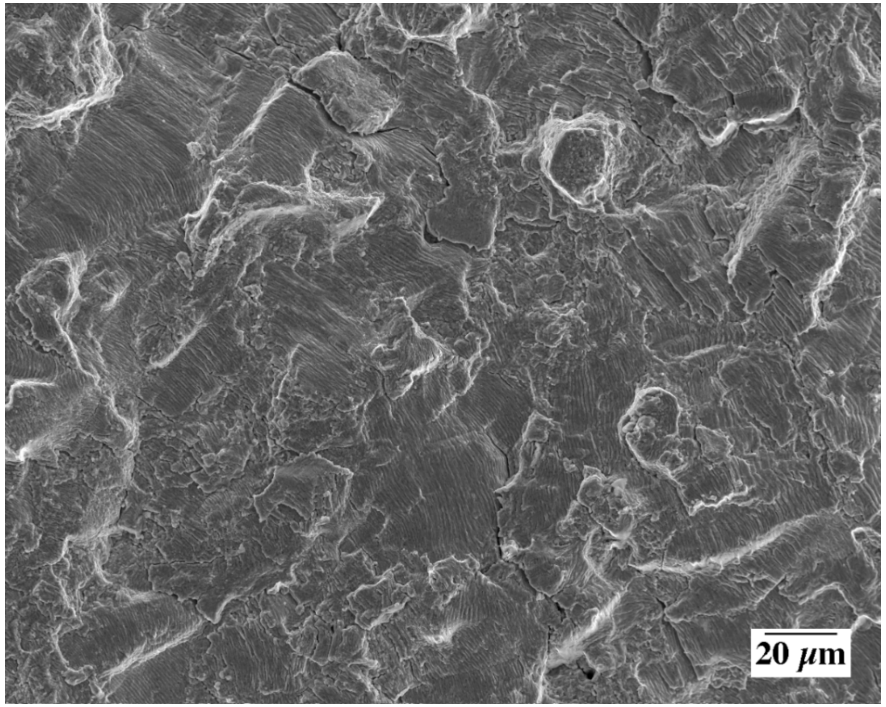
Fracture at Failure: 550°C

- Although more accumulated creep strain in the aged condition (more cycles), fracture is transgranular
- Cross-slip results in enhanced plasticity adjacent to grain boundaries, increasing local grain boundary toughness, retards propagation of the creep damage

SA condition: Intergranular Fracture,
$\sum \boldsymbol{N_f \varepsilon_{cr}} = \mathbf{0.16}$, *$N_f$ = 974 cyc.*



Aged condition: Transgranular Fracture,
$\sum \boldsymbol{N_f \varepsilon_{cr}} = \mathbf{0.68}$, *$N_f$ = 3337 cyc.*



Porter *et al.*, unpublished research

Summary and Conclusions

- At 550 °C, slip is primarily planar **in the SA condition** due to a strong inhibition of cross-slip, which prevents recovery, leads to high cyclic stresses, and rapid propagation of grain boundary damage.
- At 650 °C, the deformation character changes from planar to wavy **in the SA condition**, resulting in dynamic recovery, lower tensile stresses, and blunting of the internal damage.
- Microstructural instability of SA Alloy 709 results in an evolution of precipitate and solution strengthening in short term creep-fatigue tests. The evolution is temperature dependent!
- Dynamic precipitation decreases solute content, lowers friction stress on partials, and leads to a transition in slip character from planar to wavy.
- Static aging prior to testing consumes C and N in carbides and nitrides, promotes cross-slip at relatively low stresses, and results in an increase in creep-fatigue life compared to the SA condition.
- Increased cyclic plasticity by cross-slip retards propagation of grain boundary damage and results in transgranular fracture at 550 °C.
- Creep-fatigue performance during accelerated testing from SA condition is not representative of the expected performance during service. Deformation behavior in a pre-aged microstructure at 550 °C, which results in slow propagation of a small amount of creep damage, is more indicative of the behavior after long exposure to service temperatures.

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