Development of a Physically-Based Creep Model Incorporating ETA Phase Evolution for Nickel-Base Superalloys

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Project Objectives

The primary objective of this program is to develop a

physically based creep model

for Nimonic 263 that synthesizes known creep behavior based on gamma prime strengthening with a

new understanding of the effects of eta phase

on creep performance at long service times in fossil energy power plants.
A-USC Steam Power Generation Plants

Steam temperature: 750°C (~1400°F)
Stein Pressure: 35MPa (5000 psi)

High Temperature
Corrosive Environments
Long Service Life (>15 years)
One candidate material is Nimonic 263

- Nickel Superalloy
- Easy to form and weld
- Excellent corrosion/oxidation resistance
- Good creep performance
- Strengthening mechanisms
  - Precipitate strengthening
  - Solid solution strengthening
- Other common materials include Haynes 282 and Inconel 740

<table>
<thead>
<tr>
<th>Element</th>
<th>Percent</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ni</td>
<td>48</td>
</tr>
<tr>
<td>Co</td>
<td>20</td>
</tr>
<tr>
<td>Cr</td>
<td>20</td>
</tr>
<tr>
<td>Mo</td>
<td>6</td>
</tr>
<tr>
<td>Ti</td>
<td>2</td>
</tr>
<tr>
<td>Al</td>
<td>0.60</td>
</tr>
<tr>
<td>Fe</td>
<td>0.70</td>
</tr>
<tr>
<td>Mn</td>
<td>0.60</td>
</tr>
<tr>
<td>Si</td>
<td>0.40</td>
</tr>
<tr>
<td>C</td>
<td>0.06</td>
</tr>
</tbody>
</table>
N263 - Typical microstructure

\( \gamma \) matrix (fcc)
- Solid solution strengthened phase

\( \gamma' \) particles (L1\(_2\))
- \( \text{Ni}_3(\text{Ti,Al}) \)
- Coherent with \( \gamma \) matrix
- Precipitate strengthening phase
N263 - Typical microstructure

850°C 1 hour - SEM

750°C 50 hour - TEM

Zhao 2001
\( \gamma' \) is metastable, converts to \( \eta \) with time.

- \( \eta \) phase (DO\(_{24}\))
  - Ni\(_3\)Ti
  - Forms at the expense of \( \gamma' \)
  - Not completely coherent with \( \gamma \) matrix
  - Slow kinetics of formation
η has slow kinetics of formation.

Often ‘Turned off’ in Thermocalc simulations for initial conditions
η forms DURING creep tests.

Inconel 740 at 750°C

Start of η Phase at Grain Boundary
η Phase Evolution
η phase formed DURING test

Shingledecker, 2012
There are conflicting reports in the literature about effects of η on creep properties.

<table>
<thead>
<tr>
<th>Material</th>
<th>Temperature</th>
<th>Time</th>
<th>Comment</th>
</tr>
</thead>
<tbody>
<tr>
<td>Nimonic 263</td>
<td>800 °C</td>
<td>700 hrs</td>
<td>Reduces creep ductility; cavity nucleation and microcracking; avoid near grain boundary</td>
</tr>
<tr>
<td>Nimonic 263</td>
<td>816 - 840 °C</td>
<td>1100-1400 hrs</td>
<td>Claim detrimental to strength and ductility</td>
</tr>
<tr>
<td>Inconel 740</td>
<td>750 - 850°C</td>
<td>1000 hrs</td>
<td>Presence at grain boundaries reduced impact toughness</td>
</tr>
<tr>
<td>Inconel 740</td>
<td>816 °C</td>
<td>2500 hrs</td>
<td>Reduce γ’ strengthening/limit grain boundary ductility</td>
</tr>
<tr>
<td>Inconel 740</td>
<td>750 °C</td>
<td>2000-20000 hrs</td>
<td>Not detrimental to creep; formation kinetics faster under stress</td>
</tr>
<tr>
<td>Inconel 740</td>
<td>750 - 850°C</td>
<td>1000-20000 hrs</td>
<td>Reduced creep rupture ductility above 7 vol% eta</td>
</tr>
<tr>
<td>Inconel 740</td>
<td>750 °C</td>
<td>2000-23000 hrs</td>
<td>Not detrimental to creep</td>
</tr>
</tbody>
</table>

Michigan Technological University | EPRI | U.S. Department of Energy
Problem statement

• η phase **will form** in A-USC components in service
• There is **no agreement** in the literature about whether η phase is detrimental to creep performance
• There has been **no research** about how η phase might affect constitutive behavior (creep rates), and therefore life prediction
• η phase might also affect cavitation behavior
We aim to study the effects of $\eta$ on creep properties of Nimonic 263

- Study creep deformation and failure mechanisms varying across the microstructural spectrum

1. **Material 1**  
   - $\gamma'$ only  
   - Standard N263  
   - Contains $\gamma'$ only, prior to creep test

2. **Material 2**  
   - $\gamma'$ + $\eta$  
   - Standard N263  
   - Heat treated prior to creep test to contain $\gamma'$ and $\eta$

3. **Material 3**  
   - $\eta$ only  
   - Modified Michigan Tech alloy that contains $\eta$ only, prior to creep test

Michigan Technological University
Material 1 – γ’ only

- Standard Commercial Nimonic 263
- Widely studied
- Creep data available from an earlier research carried out by EPRI
- Crept specimens from EPRI available for deformation studies
Material 3 – $\eta$ only

- Earlier Michigan Tech research - Goal to create Nimonic 263 with all $\eta$ and no $\gamma'$
- DOE Approach using Thermo-Calc

<table>
<thead>
<tr>
<th>Alloy Element</th>
<th>Ni</th>
<th>Cr</th>
<th>Co</th>
<th>Ti</th>
<th>W</th>
<th>Nb</th>
<th>Ta</th>
<th>V</th>
<th>Fe</th>
<th>Mn</th>
<th>Al</th>
<th>C</th>
<th>Mo</th>
</tr>
</thead>
<tbody>
<tr>
<td>Nimonic 263</td>
<td>Bal</td>
<td>19.8</td>
<td>19.9</td>
<td>2.10</td>
<td>0.16</td>
<td>0.01</td>
<td>0</td>
<td>0.01</td>
<td>0.40</td>
<td>0.39</td>
<td>0.47</td>
<td>0.06</td>
<td>5.93</td>
</tr>
<tr>
<td>Alloy 20</td>
<td>Bal</td>
<td>20.8</td>
<td>20.7</td>
<td>2.75</td>
<td>1.94</td>
<td>1.92</td>
<td>1.09</td>
<td>0.85</td>
<td>0.48</td>
<td>0.42</td>
<td>0.14</td>
<td>0.07</td>
<td>0.01</td>
</tr>
</tbody>
</table>
Material 3 aged at 850°C
Creep tests were carried out from 700°C to 850°C.
Material 2 – γ’ + η

• Performed simulations in ThermoCalc with η phase ‘on’ and ‘off’ to work around sluggish η phase formation

• Conducted Literature review for experimental findings of phase formations to supplement ThermoCalc

• Samples were heat treated at 750°C, 800°C, 850°C, 900°C for 100hr, 500hr, 1000hr, 5000hr
η volume fraction and γ’ particle size vs time

750 °C – Very little η develops in these times
900°C – Near solvus temperature, most γ’ and η has dissolved
We chose 850°C 1000H heat treatment for Material 2
Creep tests

Characterization

Modelling
Predicted rupture time (days)

~500 days of creep tests planned
~170 days considering 3 frames
Should be done by October
Considering stress change creep tests
Creep tests at 700°C 200MPa

Material 1
Creep tests at 700°C 200MPa

Material 2
Ongoing

Material 3
220MPa*

Creep vs time
Creep tests at 700°C 400MPa

**Creep vs time**

![Graph showing creep vs time for Material 1 and Material 3.](image)

- Material 1
- Material 3
Creep tests at 800C 145MPa

Creep vs time

Material 3
Material 2
Ongoing
Creep tests at 800°C 250MPa

Creep vs time

Time (hrs)

Creep (%)
Creep tests - preliminary summary

- Creep properties (first glance) – 1 > 2 > 3
- Potential reasons:
  - Presence of $\eta$, and/or
  - Coarsened $\gamma'$, and/or
  - Reduced $\gamma'$ volume fraction
- Requires further experimentation and modelling
TEM studies to identify deformation mechanisms

Characterization to identify phases, fractions

Deformation mechanism maps

Creep model
Choosing a baseline creep model for γ’ strengthened Nickel superalloys

- Substantial prior research has been conducted by many investigators to develop physically-informed creep models for these types of alloys. (Dyson et al., many others)
- DOE-sponsored research by Shen Chen and his team at GE Global Research resulted in an outstanding model that worked very well for Haynes 282
  - DE-FE0005859 and DE-FE0024027

Shen Chen 2014
Creep model

• Chen Shen’s code based on Dyson model used at starting point

• His material - Haynes 282 with $\gamma'$ (no $\eta$)

• The model includes microstructural parameters such as $\gamma'$ size and volume fraction, APB energy, $\gamma'$ coarsening in service, diffusional parameters, etc.

• The output of the code is plot of creep strain vs time for given input temperature, stresses, variables and precipitate coarsening data over time. *Includes cavitation and failure.*
\[ \varepsilon_{\text{creep}} = \varepsilon_{\text{dislocation}} + \varepsilon_{\text{diffusion}} \]

\[ \varepsilon_{\text{dislocation}} = \varepsilon_{\text{climb}} + \varepsilon_{\text{shearing}} \]

\[ \varepsilon_{\text{disloc}} = \left\{ \begin{array}{ll} \rho A f (1 - f) \left( \frac{\pi}{4f} - 1 \right) \sinh \left( C \frac{\sigma_{\text{eff}} - \sigma_B - \sigma_0}{M k T} b^2 \lambda \right) & \text{if } \sigma_{\text{eff}} - \sigma_B - \sigma_0 > 0 \\ 0 & \text{otherwise} \end{array} \right. \]

\[ \sigma_{\text{shear}} = \frac{Y_{APB}}{2b} \left[ \left( \frac{12 Y_{APB} f r}{\pi G b^2} \right)^{\frac{1}{2}} - f \right] \]

\[ \sigma_{\text{climb}} = \frac{2f}{1 + 2f} \sigma_{\text{eff}} \left[ 1 - \exp \left( - \frac{1 + 2f}{2(1 - f)} \frac{E \varepsilon_{\text{disloc}}}{\sigma_{\text{eff}}} \right) \right] \]
Code development

Chen Shen’s model

Material parameters hardcoded in MATLAB model

Precipitate coarsening handled by a look-up table and interpolation

Our model

Implemented a Graphical User Interface (GUI) that allows the user to change important variables

Changed code to allow input of an LSW precipitate coarsening model
Temperature Stress

Temperature (degree C) 760  Stress (MPa) 300

Material parameters

<table>
<thead>
<tr>
<th>Material parameters</th>
<th>Fitted coefficients</th>
<th>Ostwald parameters</th>
</tr>
</thead>
<tbody>
<tr>
<td>Antiphase boundary energy</td>
<td>0.15</td>
<td></td>
</tr>
<tr>
<td>Boundary diffusion parameter</td>
<td>0.5</td>
<td></td>
</tr>
<tr>
<td>Burgar's vector</td>
<td>2.54</td>
<td></td>
</tr>
<tr>
<td>Dislocation density</td>
<td>80</td>
<td></td>
</tr>
<tr>
<td>Grain size</td>
<td>150</td>
<td></td>
</tr>
<tr>
<td>Lattice diffusion parameter</td>
<td>0.0007</td>
<td></td>
</tr>
<tr>
<td>Surface energy</td>
<td>2</td>
<td></td>
</tr>
<tr>
<td>Taylor factor</td>
<td>3.07</td>
<td></td>
</tr>
<tr>
<td>Void Grain diameter ratio</td>
<td>0.1</td>
<td></td>
</tr>
</tbody>
</table>

Material parameters

Calculate when all data entered

Strain vs time

<table>
<thead>
<tr>
<th>Stress (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>150.0 MPa</td>
</tr>
<tr>
<td>200.0 MPa</td>
</tr>
<tr>
<td>225.0 MPa</td>
</tr>
<tr>
<td>250.0 MPa</td>
</tr>
<tr>
<td>300.0 MPa</td>
</tr>
</tbody>
</table>
Summary

• Aim - To quantify effects of η phase on Creep properties of Nimonic 263

• Compare three microstructures, (all γ’) - (γ’ + η) - (all η)

• Creep tests indicate increased strain rates from \( 1 \) – \( 2 \) – \( 3 \)
Summary

• Study deformation and damage mechanisms using TEM

• Create Deformation Mechanism Maps for three microstructures

• Modify existing creep model to incorporate \( \eta \) phase effects
<table>
<thead>
<tr>
<th>Milestone Title/Description</th>
<th>Planned Completion Date</th>
<th>Actual Completion Date</th>
</tr>
</thead>
<tbody>
<tr>
<td>2.0 Develop heat treatments to form $\gamma'$ and $\eta$ phases in Nimonic 263 prior to creep testing</td>
<td>1/31/2017</td>
<td>3/1/2018</td>
</tr>
<tr>
<td>2.1 Mine existing data from the literature. If insufficient, conduct simulations with Thermo-Calc and kinetics software to predict $\eta$ phase formation in reasonable amounts of time for new material. Establish best route to form $\gamma'$ such that $\gamma'$ structure is as close to standard Nimonic 263 as possible.</td>
<td>11/30/2016</td>
<td>3/1/2018</td>
</tr>
<tr>
<td>2.2 Validate predictions in (2.1) experimentally, and adjust as needed.</td>
<td>1/31/2017</td>
<td>6/15/2018</td>
</tr>
<tr>
<td>Critical Decision Point. Is it possible to produce a suitable $\gamma'$ + $\eta$ microstructure via a relatively short time (&lt; 1,000 hour) heat treatment? If yes, continue. If not, see Section B, Risk Management, for mitigation strategies.</td>
<td>1/31/2017</td>
<td>12/22/2017</td>
</tr>
<tr>
<td>3.0 Conduct creep tests at EPRI on new Nimonic 263 that had been modified to contain both $\gamma'$ and $\eta$ phases.</td>
<td>8/31/2018</td>
<td>25%</td>
</tr>
<tr>
<td>Milestone Title/Description</td>
<td>Planned Completion Date</td>
<td>Actual Completion Date</td>
</tr>
<tr>
<td>-------------------------------------------------------------------------------------------</td>
<td>-------------------------</td>
<td>------------------------</td>
</tr>
<tr>
<td>4.0 Assess microstructures as well as deformation and damage mechanisms in all three microstructural conditions (100% γ’, 100% η, mixture of γ’ + η.)</td>
<td>2/28/2019</td>
<td>25%</td>
</tr>
<tr>
<td>4.1 Conduct optical, SEM and TEM microscopy to quantify phase transformations, precipitate size evolution, deformation mechanisms (TEM), and damage evolution.</td>
<td>10/31/2018</td>
<td>30%</td>
</tr>
<tr>
<td>4.2 Establish effects of microstructure on deformation mechanisms in all three microstructures</td>
<td>1/31/2019</td>
<td>0%</td>
</tr>
<tr>
<td>4.3 Use results of (4.1) and (4.2) to quantify the effects of η on creep performance of Nimonic 263.</td>
<td>2/28/2019</td>
<td>0%</td>
</tr>
<tr>
<td>5.0 Modify existing γ’ based creep models to account explicitly for the effects of η phase as determined in (4.)</td>
<td>8/31/2019</td>
<td>35%</td>
</tr>
<tr>
<td>5.1 Assess and integrate best damage models from the literature</td>
<td>2/28/2019</td>
<td>60%</td>
</tr>
<tr>
<td>5.2 Adapt models to explicitly include the transformation from metastable γ’ to equilibrium η and resultant changes in damage mechanisms</td>
<td>6/30/2019</td>
<td>0%</td>
</tr>
<tr>
<td>5.3 Validate model with select creep experiments</td>
<td>8/31/2019</td>
<td>0%</td>
</tr>
</tbody>
</table>
Questions?

TEM studies to identify deformation mechanisms

Characterization to identify phases, fractions

Deformation mechanism maps

Creep model