

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

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2018 Annual Review Meeting for Crosscutting Research; 04/10/2018

DE-FE-0027822: Performance period 8/15/2016 – 3/31/2018



Michigan
Technological
University



U.S. DEPARTMENT OF
ENERGY



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

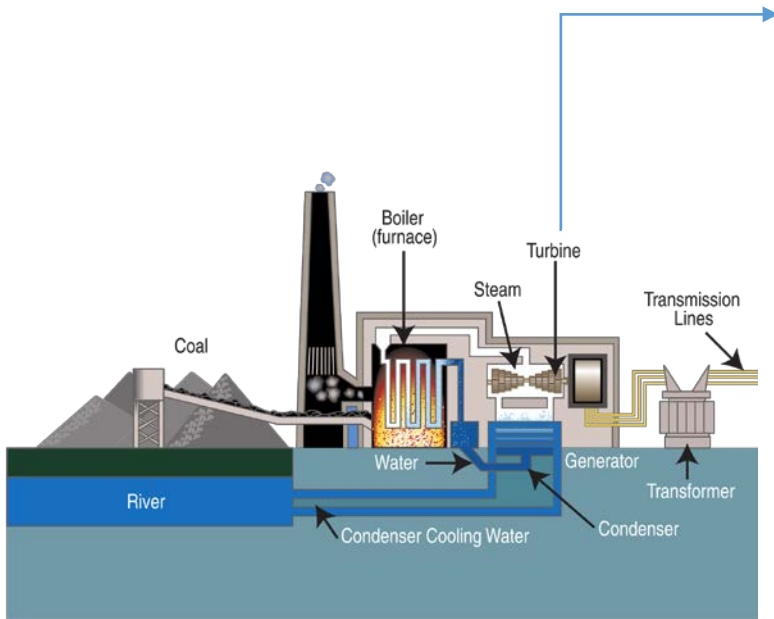
new understanding of the effects of eta phase

on creep performance at long service times in fossil energy power plants.

Outline

- Background
- Problem Statement
- Experimental Approach
- Results
- Conclusion

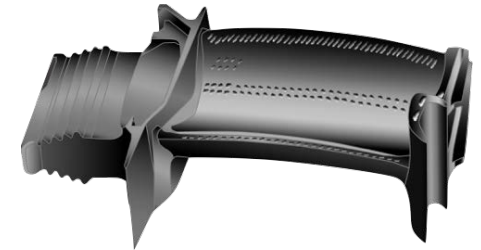
1. Background



Conventional Fossil-Fired Steam Power Plant
Nuclear and Combined Cycle Power Plant



Steam Turbine



Turbine Blade

- High Temperature
- Corrosive Environments
- Long Service Life

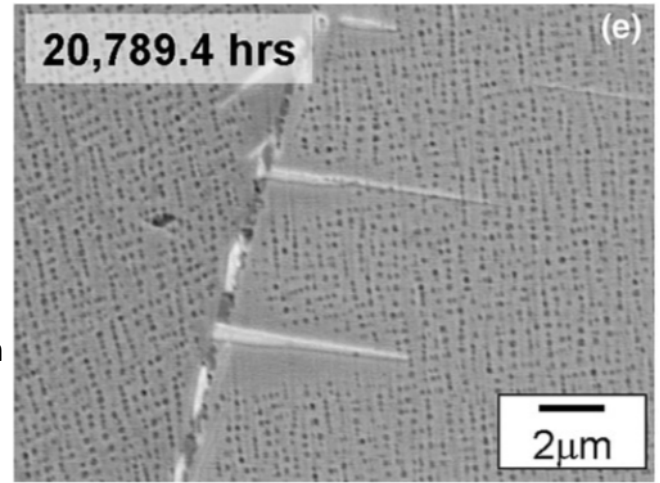
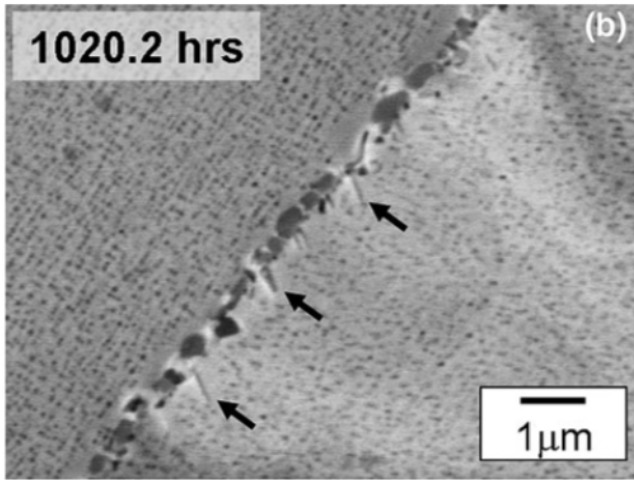
Background – Nimonic 263

- Nickel–base Superalloy
- Excellent corrosion/oxidation resistance
- Good creep performance
- Easy to form and weld (Low volume fraction of γ')
- Candidate material for A-USC piping and other components

Ni	Co	Cr	Al	Ti	Mo	Fe	Mn	Si	C
48	20	20	0.60	2	6	0.70	0.60	0.40	0.06

Background – Nimonic 263

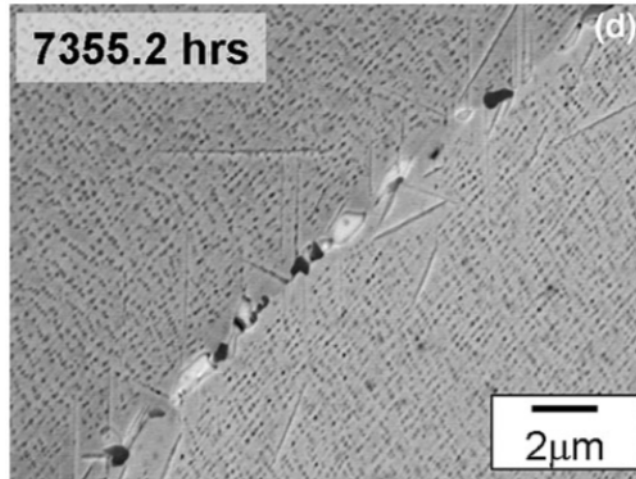
- Over long service life and at high temperatures, η phase is known to form at the expense of γ' phase
- Previous creep studies on Nimonic 263 and similar alloys have shown growth of η phase **during the course of creep tests**



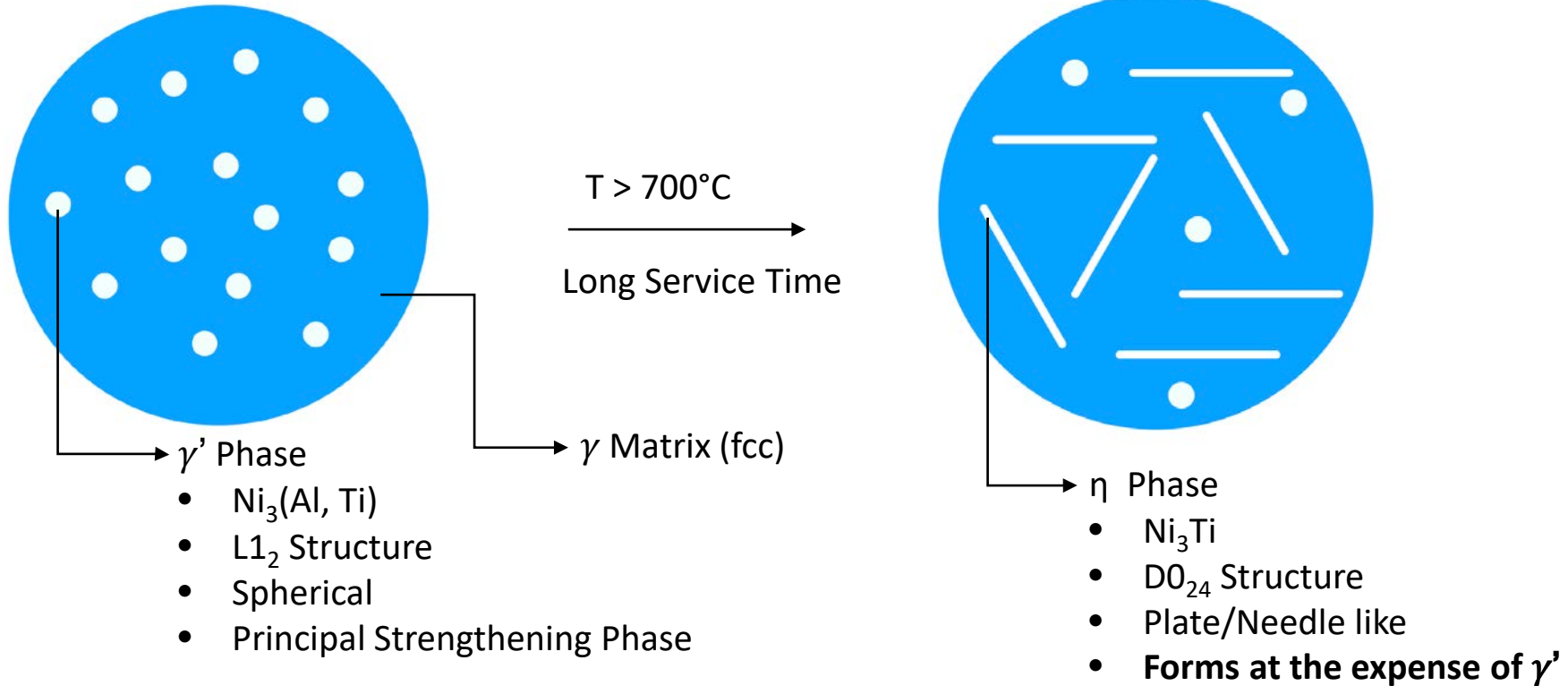
Eta Phase evolution

- Gamma Prime Particles
- Start of Eta Phase at Grain Boundary

Inconel 740 750°C
[Shingledecker and Pharr 2012]



Nimonic 263 Evolution



Conflicting Reports from Literature about η Phase

Nimonic 263 [Zhang 2002]	800 °C	700 hrs	Reduces creep ductility; cavity nucleation and microcracking; avoid near grain boundary
Nimonic 263 [Zhao 2002]	816-840 °C	1100-1400 hrs	Claim detrimental to strength and ductility
Inconel 740 [Zhao 2003]	750-850 °C	1000 hrs	Presence at grain boundaries reduced impact toughness
Inconel 740 [Evans 2004]	816 °C	2500 hrs	Reduce γ' strengthening/limit grain boundary ductility
Inconel 740 [Shingledecker 2012]	750 °C	2000-20000 hrs	Not detrimental to creep; formation kinetics faster under stress
Inconel 740 [Shingledecker 2013]	750-850 °C	1000-20000 hrs	Reduced creep rupture ductility above 7 vol% eta
Inconel 740 [Unocic 2014]	750 °C	2000-23000 hrs	Not detrimental to creep

2. Problem Statement

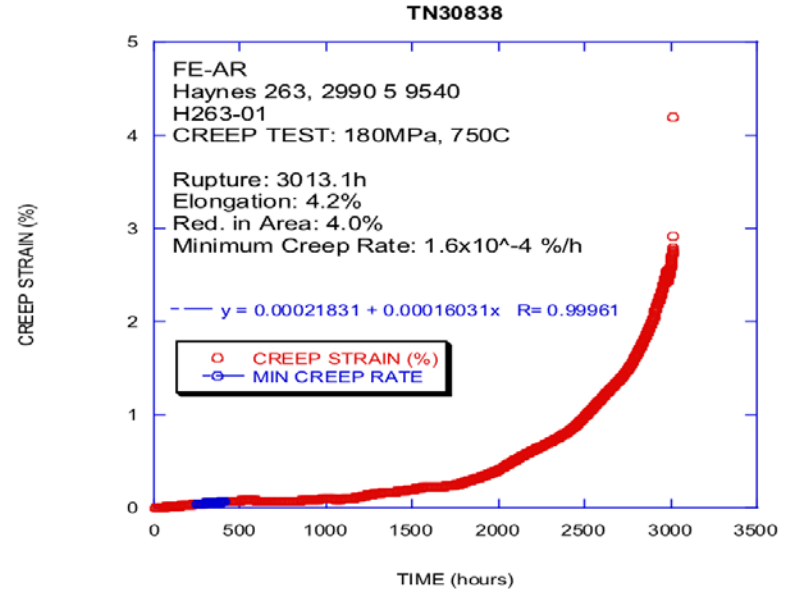
- η phase **will form** in A-USC components in service
- There is **no agreement** in the literature about whether η phase is detrimental to creep performance
- There has been **no research** about how η phase might affect constitutive behavior (creep rates), and therefore life prediction
- η phase might also affect cavitation behavior

3. Experimental Approach

- Want to isolate effects of η phase on Creep performance
- Compare creep performance and deformation mechanism of three materials:
 - Material 1 (γ' only) - Standard Commercial Nimonic 263 containing only γ'
 - Material 2 (η only) - A modified Michigan Tech alloy based on Nimonic 263 that contains no γ' , only η
 - Material 3 ($\gamma' + \eta$) - Standard Commercial Nimonic 263 that has been heat treated prior to creep test to contain both γ' and η

Material 1: Nimonic 263 - γ' only

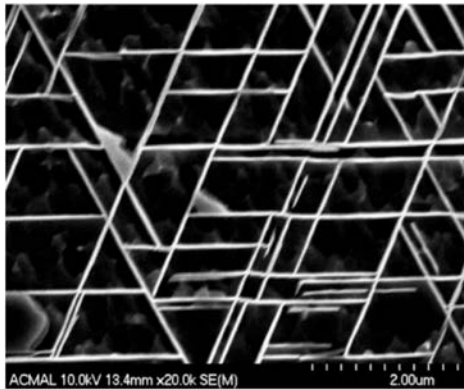
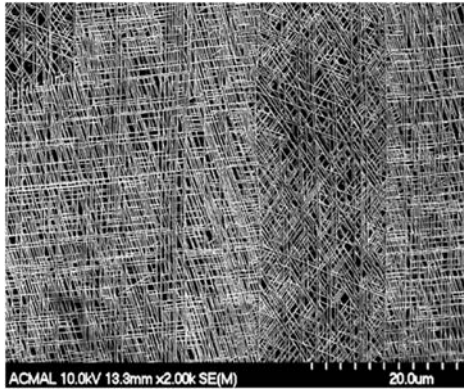
- Widely studied
- Creep data available from an earlier research carried out by EPRI
- Crept specimens from EPRI available for deformation studies



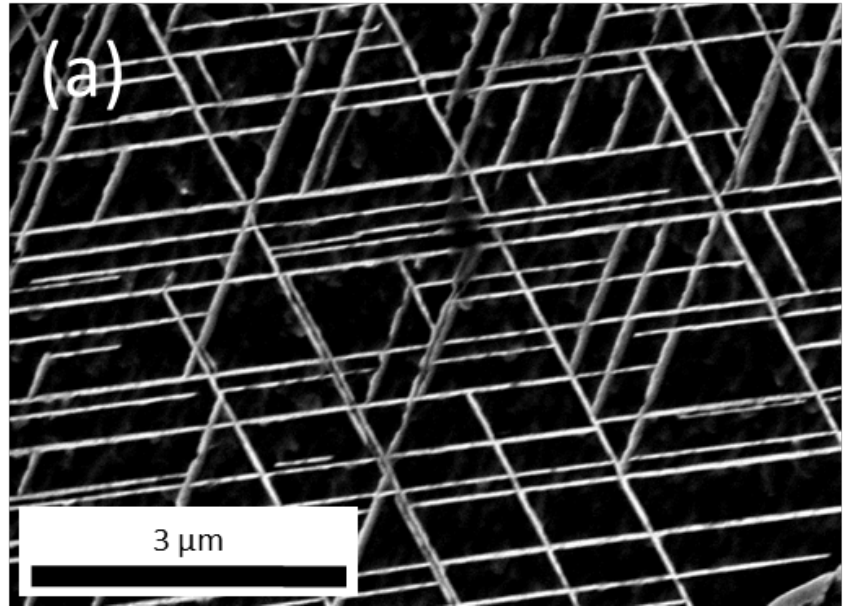
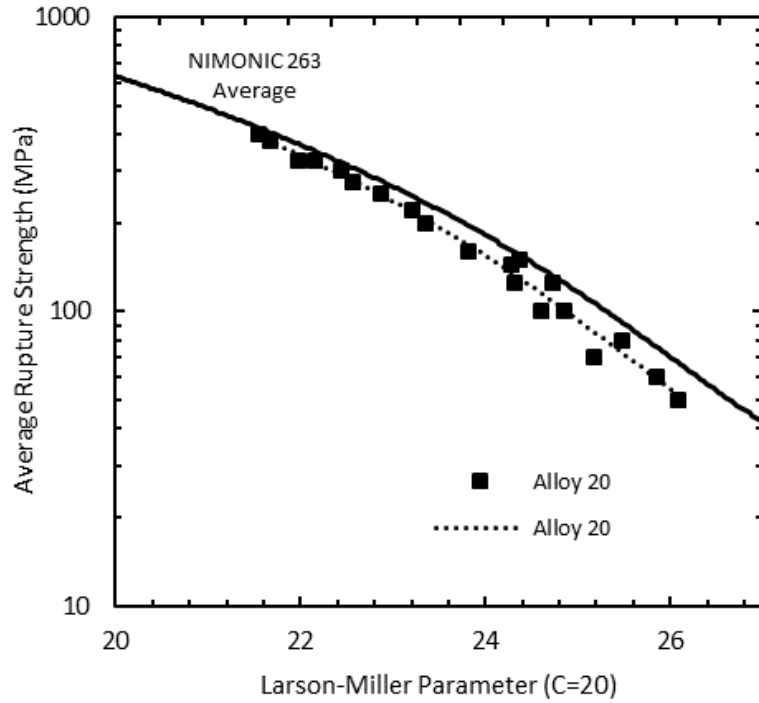
Material 2: Modified Nimonic 263 based alloy - η only

- Earlier Research – Goal to design alloys containing only η and no γ'
- DOE Approach utilizing Thermocalc was used with Nimonic 263 as starting point
- Out of 32 combinations, 3 alloys were produced and fabricated
- Lower Al, Mo and higher Ti, Nb, Ta and W (than N263) formed essentially only η and no γ'
- Creep rupture tests were conducted from 700 °C – 850 °C
- Larson Miller Parameter was plotted against rupture strength, and deformation mechanisms were determined

Modified Michigan Tech η Alloy



Alloy Element	Al	Co	Cr	Fe	Mn	Mo	Nb	Ni	Ta	Ti	V	W	C
NIMONIC 263	0.47	19.9	19.8	0.40	0.39	5.93	0.01	Bal	0	2.10	0.01	0.16	0.06
Alloy 20	0.14	20.7	20.8	0.48	0.42	0.01	1.92	Bal	1.09	2.75	0.85	1.94	0.07



Big Picture

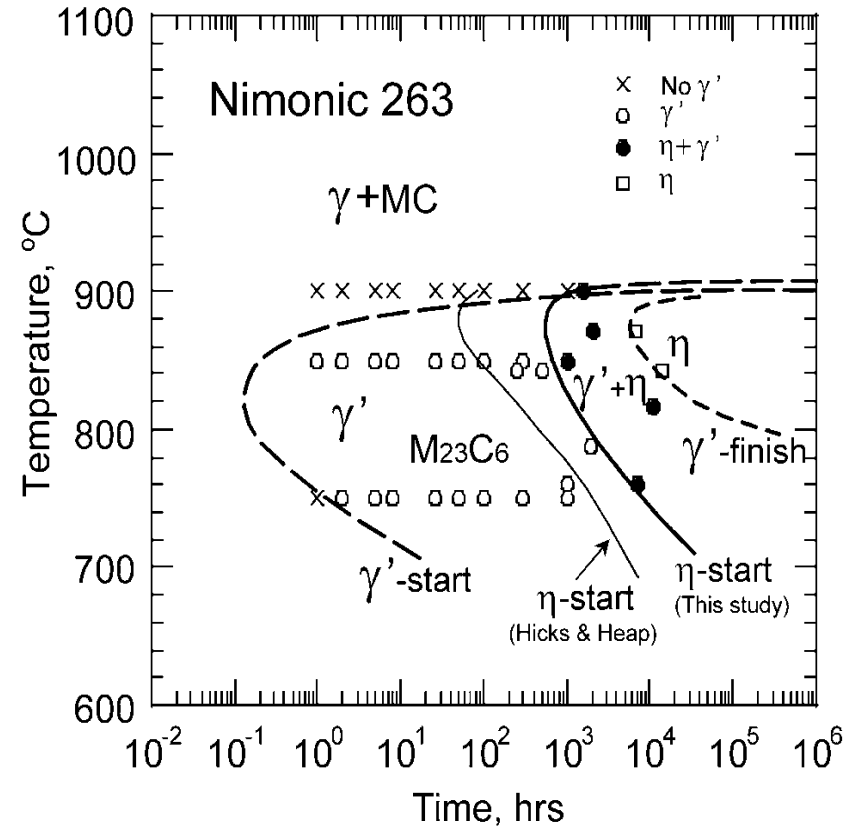
	Material 1	Material 2	Material 3
Microstructure Target	All γ'	All η	$\gamma' + \eta$ prior to creep test
Thermal Processing	Commercial	Heat treat to form η	This Project
Creep Data available	✓	✓	
Crept Specimen Available?	✓	✓	

Overview: Material 3 (Nimonic 263 γ' + η)

- Develop Heat treatment for Standard Commercial Nimonic 263 to contain γ' and η prior to creep test
- Study Creep Deformation and Failure mechanisms in:
 - This material, containing γ' and η prior to creep test
 - Standard Nimonic 263 containing only γ' prior to creep test
 - The alloy containing only η
- Modify existing creep models to incorporate deformation mechanisms of all three materials

Material 3: Nimonic 263 with γ' + η

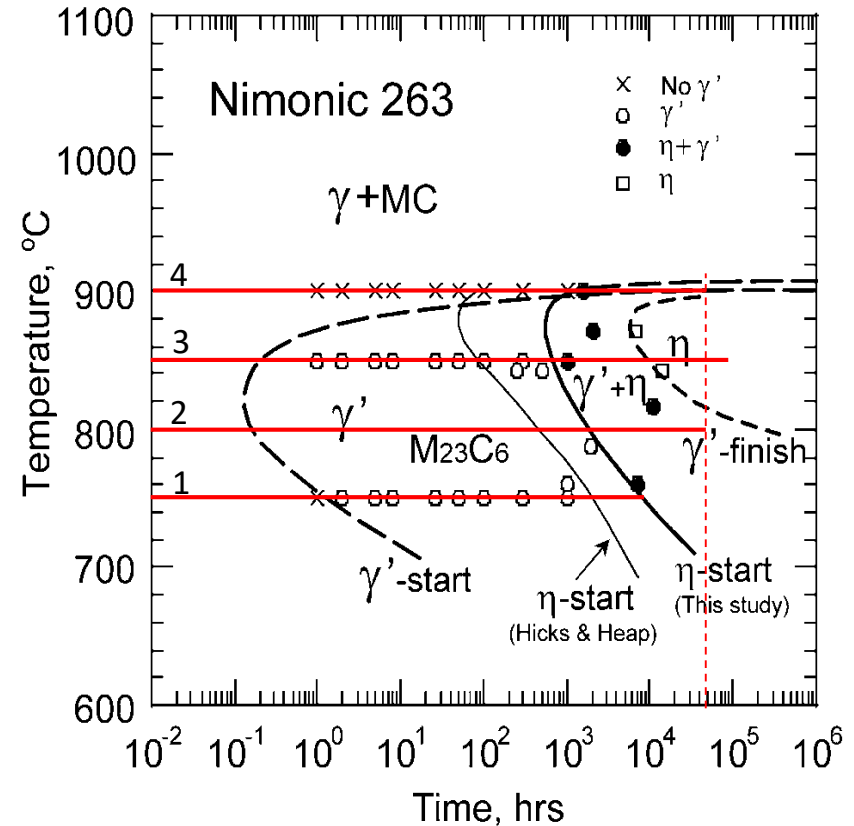
- Performed simulations in ThermoCalc with η phase 'on' and 'off' to work around sluggish η phase formation
- Conducted Literature review for experimental findings of phase formations to supplement ThermoCalc
- Samples were heat treated at 750°C, 800°C, 850°C, 900°C for 100hr, 500hr, 1000hr, 5000hr



Zhao et al., 2001

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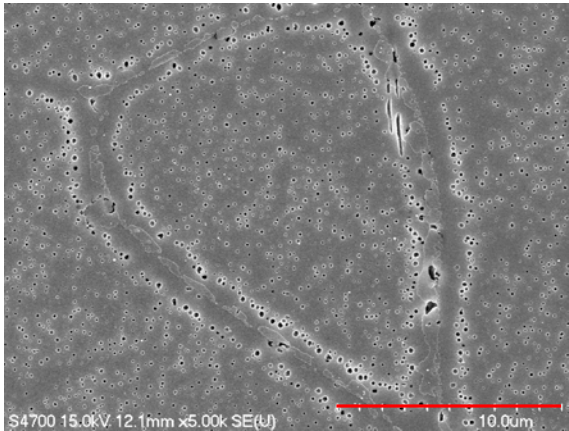


Zhao et al., 2001

4. Results

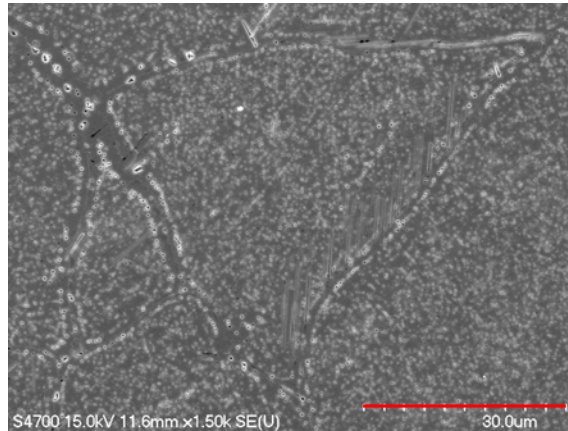
- Finished heat treatments for Material 3 (Nimonic 263 heat treated to contain $\gamma' + \eta$)
- Heat treated samples were studied with SEM to obtain volume fractions of γ' and η , as well as the particle size of γ'
- Results were validated with literature values, ThermoCalc predictions

Typical Aged Nimonic 263 - γ' and η Micrographs



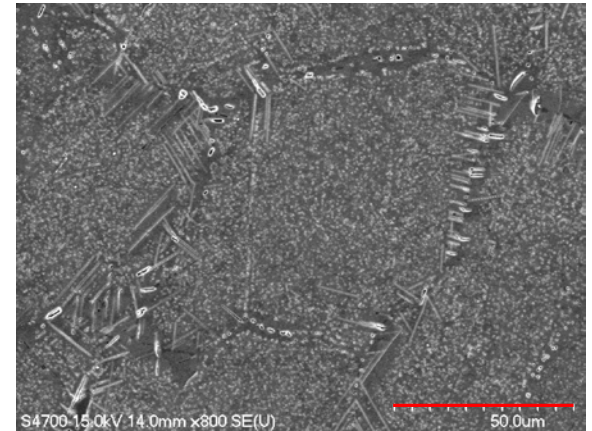
850 °C 100 hr

Scale: 10 um



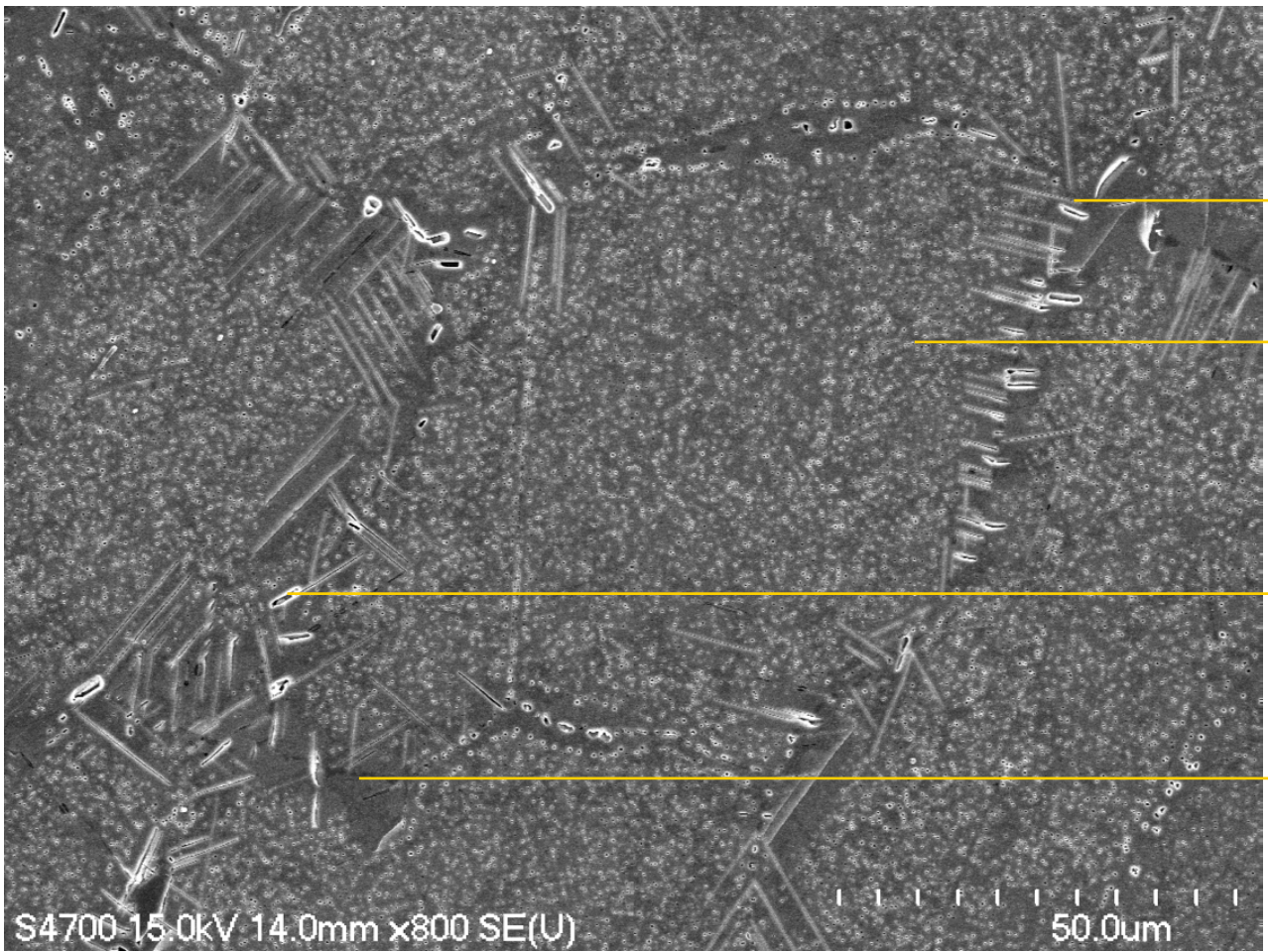
850 °C 500 hr

Scale: 30 um



850 °C 1000 hr

Scale: 50 um



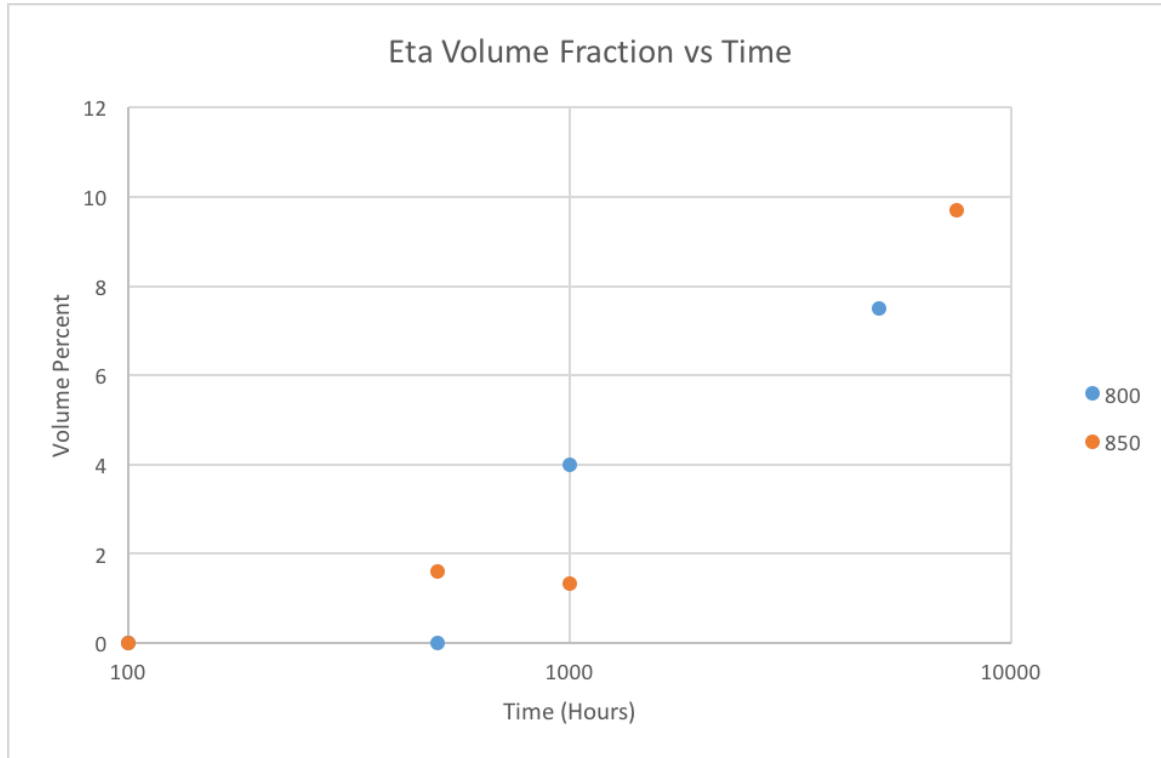
η phase (Needle/plate like)

γ' phase (Spherical)

Carbide Phases

So called γ' depletion region. Formation of η occurs at expense of γ'

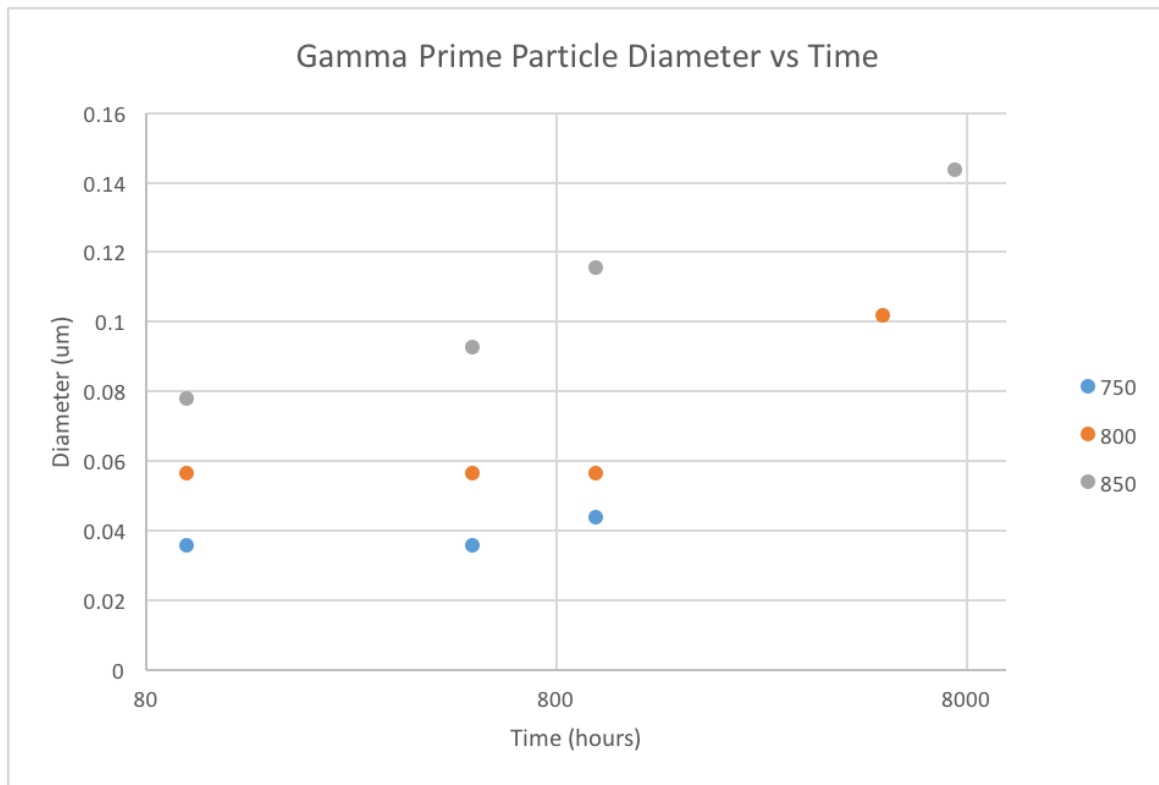
Material 3: Heat treated Nimonic 263 - η Volume Fraction



750 °C – Almost no η seen all the way till 1000 hours

900°C – Near Solvus Temperature, most γ' and η has dissolved

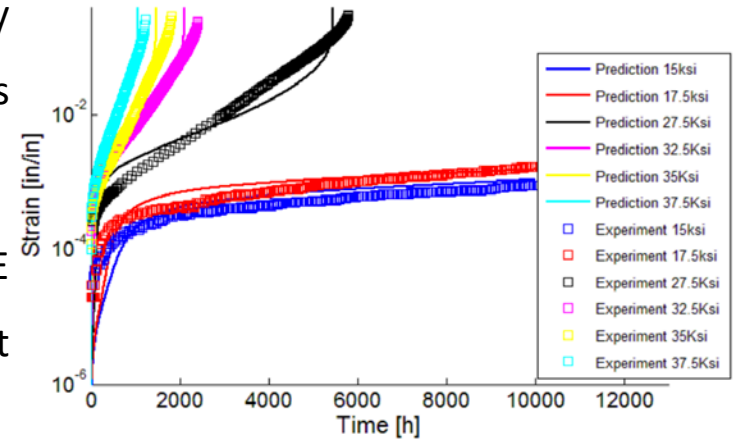
Material 3: Heat treated Nimonic 263 – γ' Particle Coarsening



Based on these results, creep specimens will be heat treated this month to contain η and γ' at the start of the creep tests

Creep Models for γ' alloys such as IN740, Haynes 282 and N263

- 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



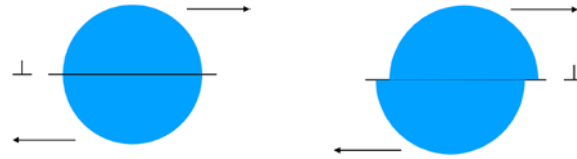
Shen Chen 2014

Creep Models for γ' alloys such as IN740, Haynes 282 and N263

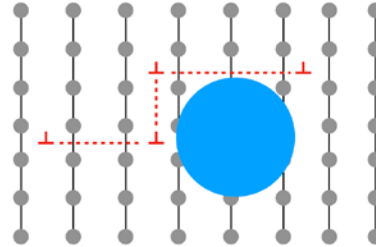
- Chen implemented a Dyson-type model in Matlab for Haynes 282.
- These models include microstructural parameters such as γ' size and volume fraction, APB energy, γ' coarsening in service, diffusional parameters, etc.
- The output of the code is plot of creep strain vs time for given input temperature, stresses, variables and precipitate coarsening data over time. ***Includes cavitation and failure.***
- Chen gave us his code, and this will be the starting point for our modelling efforts

Creep Deformation Mechanisms in Shen Model

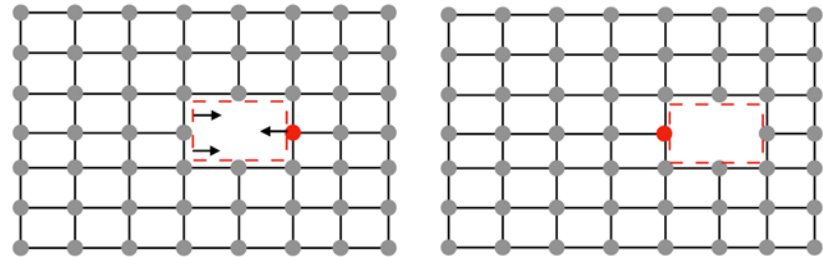
- Precipitate Shearing



- Dislocation Climb with precipitate by-pass



- Diffusional Creep (grain boundary and bulk)



Creep Model for γ'

$$\epsilon^{\text{creep}} = \epsilon^{\text{dislocation}} + \epsilon^{\text{diffusion}}$$

$$\epsilon^{\text{dislocation}} = \epsilon^{\text{climb}} + \epsilon^{\text{shearing}}$$

$$\dot{\epsilon}^{\text{diffusion}} = \dot{\epsilon}^{\text{lattice_diff}} + \dot{\epsilon}^{\text{boundary_diff}} + \dot{\epsilon}^{\text{cavity_boundary_diff}} + \dot{\epsilon}^{\text{cavity_surface_diff}}$$

Dislocation Creep Model for γ'

$$\dot{\epsilon}^{disloc} = \begin{cases} \rho A f (1 - f) \left(\sqrt{\frac{\pi}{4f}} - 1 \right) \sinh \left(C \frac{\sigma_{eff} - \sigma_B - \sigma_0}{M k T} b^2 \lambda \right) & \text{if } \sigma_{eff} - \sigma_B - \sigma_0 > 0 \\ 0 & \text{otherwise} \end{cases}$$

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

$$\sigma_{climb} = \frac{2f}{1 + 2f} \sigma_{eff} \left[1 - \exp \left(- \frac{1 + 2f}{2(1 - f)} E \frac{\dot{\epsilon}^{disloc}}{\sigma_{eff}} \right) \right]$$

Dislocation Creep Model for γ'

$$\dot{\epsilon}_{\text{diffusion}} = \dot{\epsilon}_{\text{lattice_diff}} + \dot{\epsilon}_{\text{boundary_diff}} + \dot{\epsilon}_{\text{cavity_boundary_diff}} + \dot{\epsilon}_{\text{cavity_surface_diff}}$$

$$\dot{\epsilon}_{\text{lattice_diff}} = \xi \beta \sigma_{\text{applied}} (1 + \epsilon^{\text{creep}})$$

$$\dot{\epsilon}_{\text{boundary_diff}} = 3 \pi \xi \left(\frac{1}{d}\right)^3 \sigma_{\text{applied}} (1 + \epsilon^{\text{creep}})$$

$$\dot{\epsilon}_{\text{cavity_boundary_diff}} = \xi \frac{1}{d} \frac{\sigma_{\text{applied}}}{\ln\left(\frac{1}{\tilde{\omega}_{\text{boundary diff}}}\right)}$$

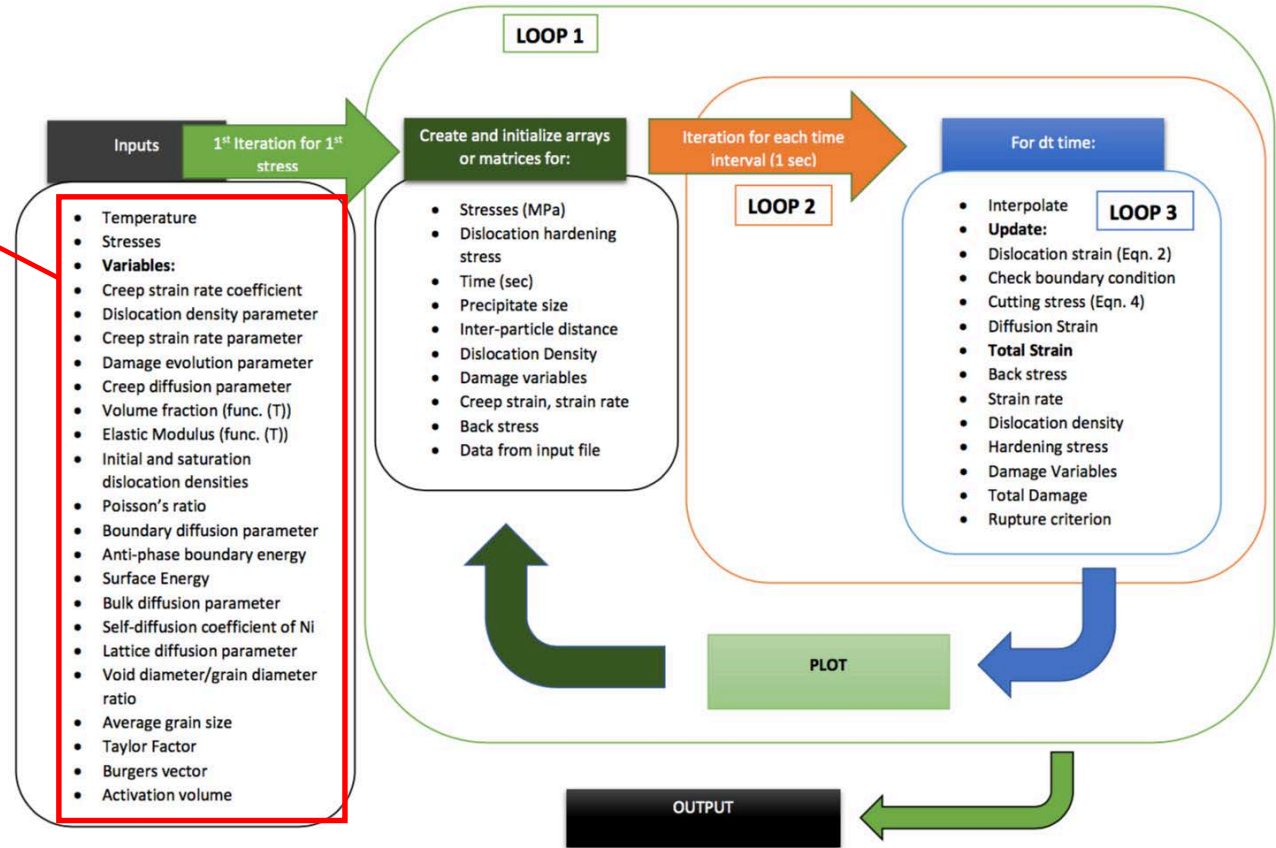
$$\dot{\epsilon}_{\text{cavity_surface_diff}} = \xi \alpha \frac{\sqrt{\tilde{\omega}_{\text{surface diff}}}}{(1 - \tilde{\omega}_{\text{surface diff}})^3} \sigma_{\text{applied}}^2$$

Code development, this project

- Chen's model is specific to Haynes 282. Material parameters are hard-coded into the Matlab files. Precipitate coarsening is handled by a look-up table and interpolation.
- To make the code usable for new alloys, and to make it easier to use, we have:
 - Implemented a GUI that allows the user to enter and quickly change all the important variables in an intuitive interface.
 - Changed the code to allow input of an LSW precipitate coarsening model in the GUI instead of hard-coded look-up tables.

MATLAB Flowchart

Define
20+
material
Parameters
 (eg. Creep
 strain rate,
 Taylor factor,
 etc)

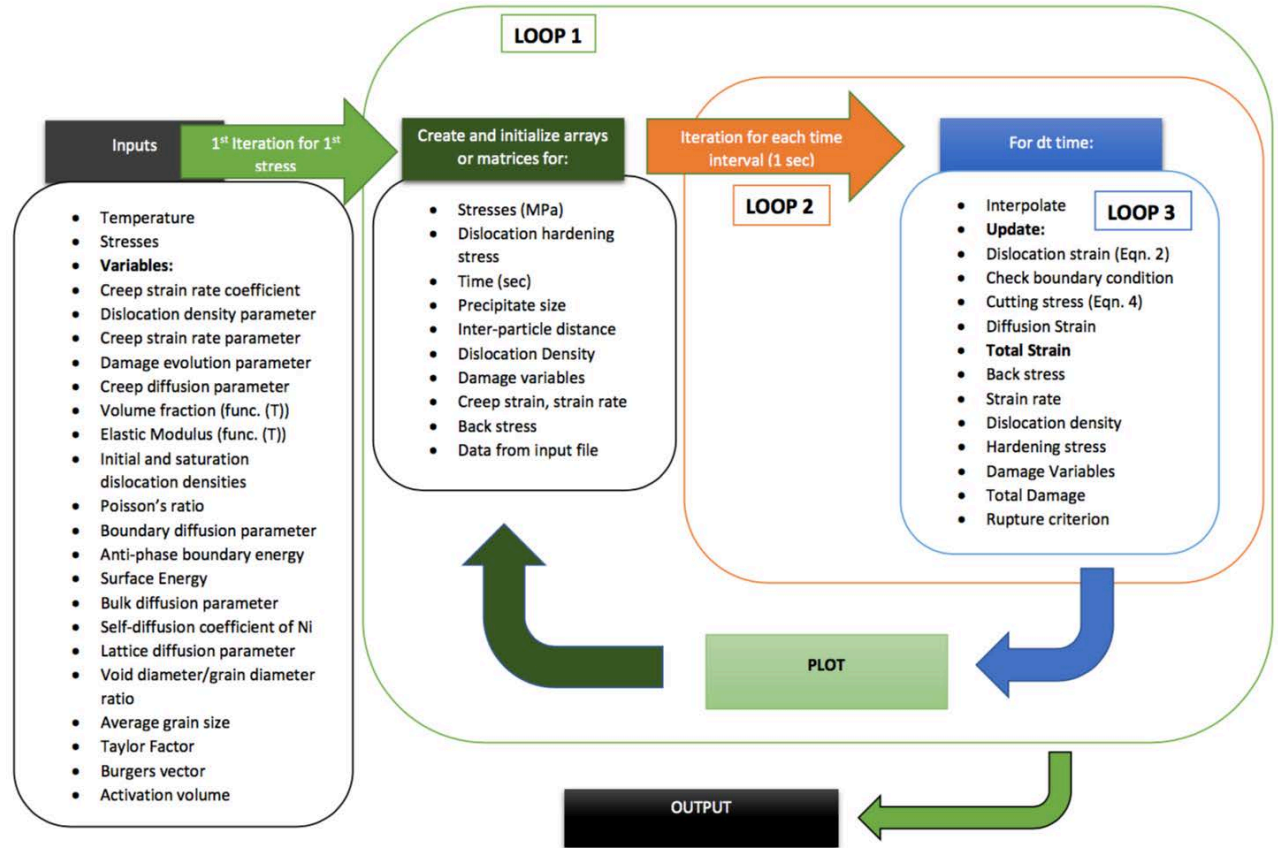


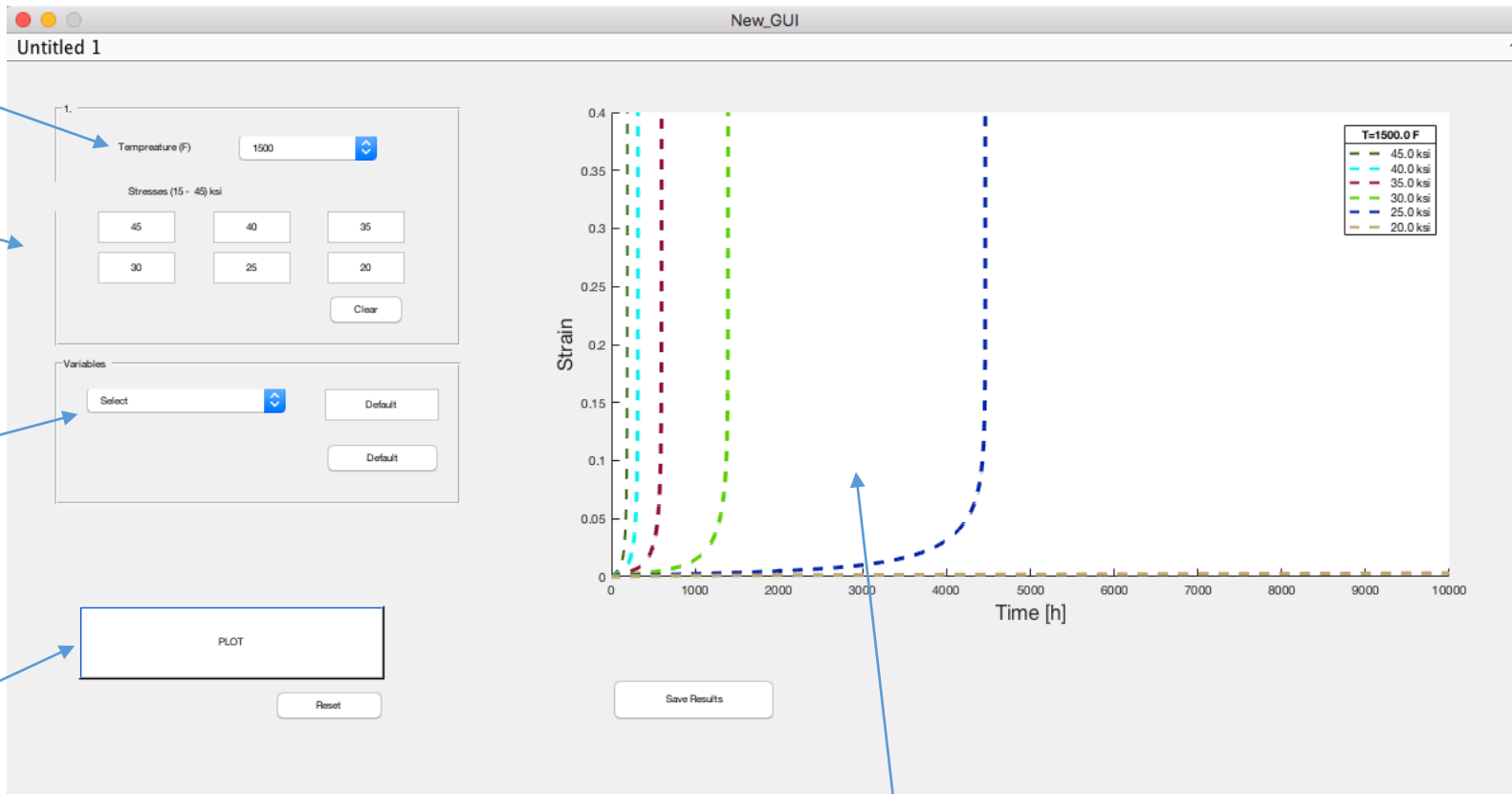
MATLAB Flowchart

Loop 1:
Initializes all variables for calculation for each stress state at $t=0$

Loop 2:
Calculates variables for time steps of 1 second

Loop 3:
Calculates strain, strain rate, damage variables and watches Rupture Criterion





Temperature

Stresses

All material parameters, via multiple drop-downs. e.g. APB energy, grain size.

Calculate when all data entered.

Predicted creep curves at whatever stresses were entered.

5. Conclusion

- Isolate effects of η in creep properties of Nimonic 263
- We have the data for Nimonic 263 with γ' and η , We have the preliminary Creep Model, now we combine
- $\gamma' + \eta$ phase: Will decide 2 heat treatments for Creep tests, this quarter
- Over next year:
 - Study Creep Deformation and Failure Mechanisms with TEM
 - Modify preliminary MATLAB model to include studies on 'all γ' ', 'all η ' and ' $\gamma' + \eta$ ' materials

Milestones

Milestone Title/Description	Planned Completion Date	Actual Completion Date
2.0 Develop heat treatments to form γ' and η phases in Nimonic 263 prior to creep testing	1/31/2017	3/1/2018
2.1 Mine existing data from the literature. If insufficient, conduct simulations with Thermo-Calc and kinetics software to predict η phase formation in reasonable amounts of time for new material. Establish best route to form γ' such that γ' structure is as close to standard Nimonic 263 as possible.	11/30/2016	3/1/2018
2.2 Validate predictions in (2.1) experimentally, and adjust as needed.	1/31/2017	95%
Critical Decision Point. Is it possible to produce a suitable $\gamma' + \eta$ microstructure via a relatively short time (< 1,000 hour) heat treatment? If yes, continue. If not, see Section B, Risk Management, for mitigation strategies.	1/31/2017	12/22/2017
3.0 Conduct creep tests at EPRI on new Nimonic 263 that had been modified to contain both γ' and η phases.	8/31/2018	20%

Milestones

Milestone Title/Description	Planned Completion Date	Actual Completion Date
4.0 Assess microstructures as well as deformation and damage mechanisms in all three microstructural conditions (100% γ' , 100% η , mixture of $\gamma' + \eta$.)	2/28/2019	15%
4.1 Conduct optical, SEM and TEM microscopy to quantify phase transformations, precipitate size evolution, deformation mechanisms (TEM), and damage evolution.	10/31/2018	10%
4.2 Establish effects of microstructure on deformation mechanisms in all three microstructures	1/31/2019	0%
4.3 Use results of (4.1) and (4.2) to quantify the effects of η on creep performance of Nimonic 263.	2/28/2019	0%
5.0 Modify existing γ' based creep models to account explicitly for the effects of η phase as determined in (4.)	8/31/2019	35%
5.1 Assess and integrate best damage models from the literature	2/28/2019	50%
5.2 Adapt models to explicitly include the transformation from metastable γ' to equilibrium η and resultant changes in damage mechanisms	6/30/2019	0%
5.3 Validate model with select creep experiments	8/31/2019	0%