THE UNIVERSITY OF TEXAS AT EL PASO
COLLEGE OF ENGINEERING

2019 Annual Review Meeting for Crosscutting Research

An Accelerated Creep Testing (ACT) Program for Advanced Creep Resistant Alloys for High Temperature Fossil Energy (FE) Applications

Robert Mach, Jacob Pellicotte, and Calvin M. Stewart

DOE Annual Review Meeting
April 10th -14th, 2019

MERG
UTEP MATERIALS AT EXTREMES RESEARCH GROUP

Outline

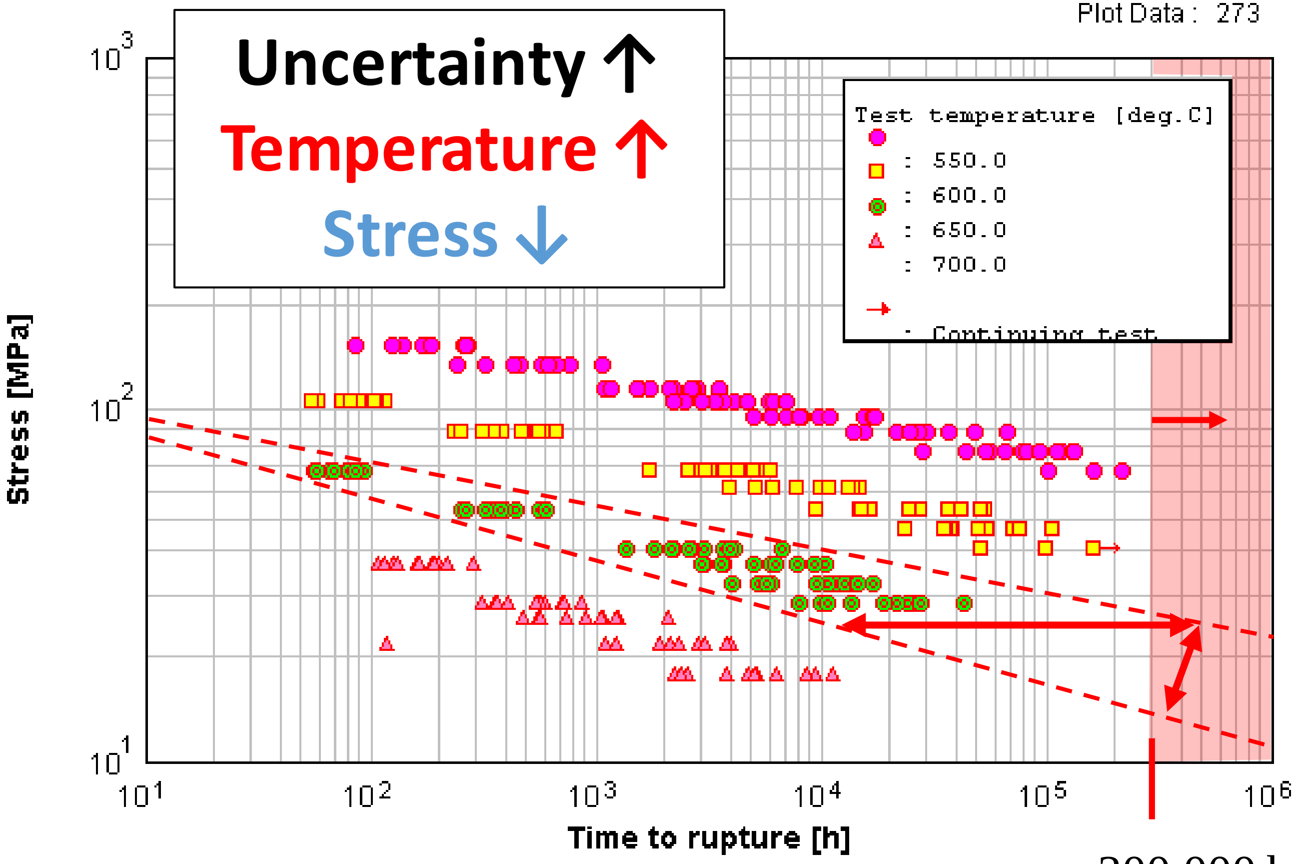
- Project Objectives
- Strategic Alignment to FE Objectives
- Technology Benchmark
- Gantt Chart
- Project Update
  - Accomplishments (Conference papers, journal articles, meetings)
  - Methodology
  - Materials and Equipment
  - Experimental Results
  - Ongoing Challenges
- Market Benefits/Assessments
- Technology-to-Market Path
- Concluding Remarks

Project Objectives

- Accelerated creep testing (ACT) is a well-established method to reduce the time-for-material-qualification; however, none of the existing ACTs provide rapid and detailed information concerning long term creep deformation and rupture behavior. Of primary concern to the FE materials scientist is the rapid experimental screening of the long-term creep behavior of candidate materials.

The **Research Objective (RO)** of this project is to vet, improve, and test the feasibility of the Stepped Isostress Method and Stress Relaxation Test for metallic materials.

Strategic Alignment



Creep-Rupture of 9Cr-1Mo Tube

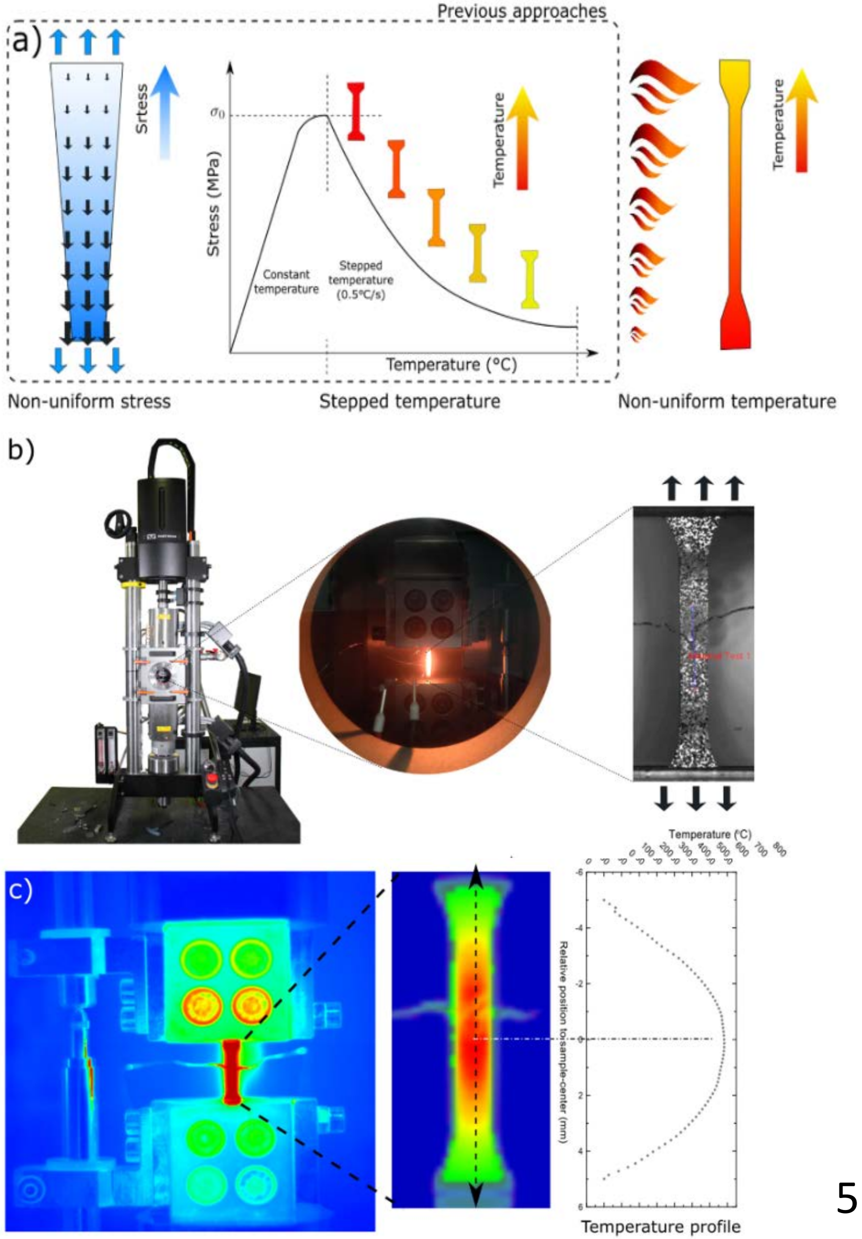
- By 2030, research and development technologies are available to support new coal-fired power plants
- The materials used in these power plants require qualification to withstand these conditions. Unfortunately, conventional creep tests are real time tests which may last up to 30 years, which is impractical for the development of power generation plants
- Accelerated creep testing, which has been proven in polymers, is a proposed method requiring only 20+ hours to gather creep deformation properties up to 10^6 hours

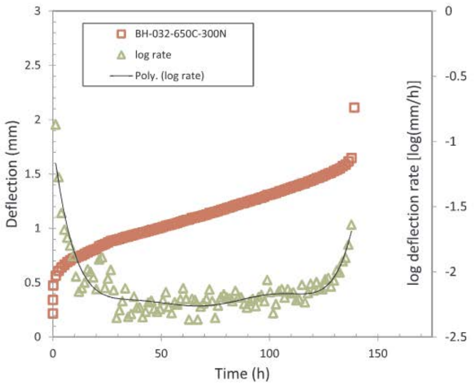
Technology Benchmark (UPDATE)

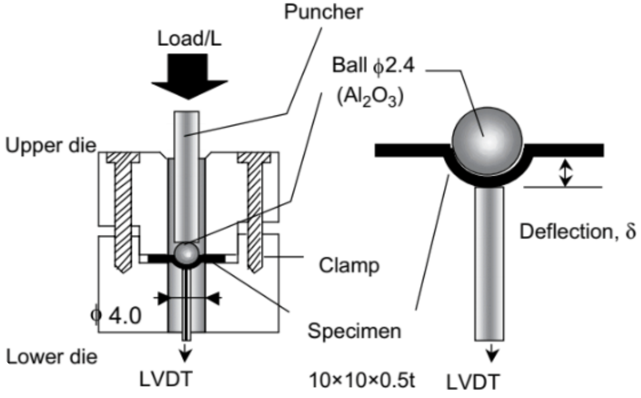
- The small punch creep (SPC) test and ultrafast creep method are current state-of-the art technology alternatives to conventional creep testing.

- An advantage for the SPC is if the material is sparse, specimens are able to be taken from components already in service

- The Ultrafast creep method is advantageous when as it accounts for aging of the specimen. The aging process is completed by treating the material in a solution at an elevated temperature followed by other various temperatures.

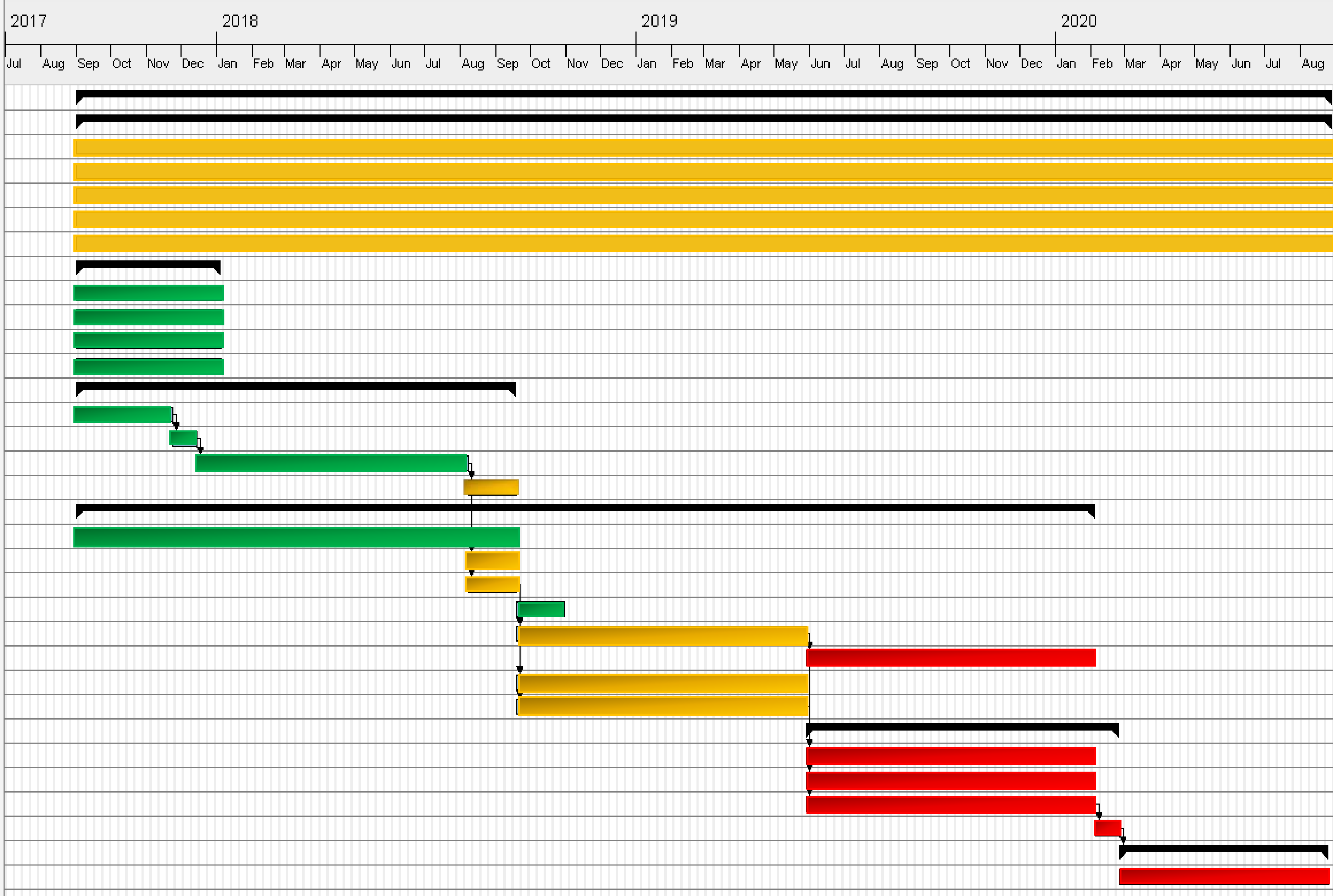






Gantt Chart

Name	Begin ...
An Accelerated Creep Testing (ACT) Program for Advance...	9/1/17
Project Management and Planning	9/1/17
Maintain Project Management Plan	9/1/17
Quarterly and Annual Reporting	9/1/17
Facility Planning	9/1/17
Maintain Data, Safety, and Quality Assurance Plan	9/1/17
Final Reporting	9/1/17
Gather Legacy Experimental Data	9/1/17
Handbooks, Theses, Dissertations	9/1/17
Journals and Proprietary Info	9/1/17
Build Digital Database	9/1/17
Sort Data using Test Information	9/1/17
Pre-ACT Mechanical Tests	9/1/17
Procure Material and Machine Specimen	9/1/17
Montonic Tensile	11/24/17
Conventional Creep Tests	12/15/17
Slow Strain Rate Tests	8/8/18
Execute SIM and SSM Tests	9/1/17
Theory Development	9/1/17
Reference-Calibration	8/8/18
Test Matrix Optimization	8/8/18
Procure Material and Machine Specimen	9/19/18
SSM Tests	9/19/18
SIM Tests	5/29/19
Improve ACT Procedure	9/19/18
Write MATLab Code	9/19/18
Post-Audit Validation	5/29/19
Credibility and Physical Realism	5/29/19
Assess Accuracy	5/29/19
Assess Precision	5/29/19
Go/No Go Decision	2/5/20
Test Standard	2/26/20
Write Standard	2/26/20



Team

Dr. Calvin M. Stewart
Associate Professor
Department of Mechanical Engineering
Principal Investigator
cmstewart@utep.edu

Robert M. Mach
Masters Research Assistant
rmmach@miners.utep.edu

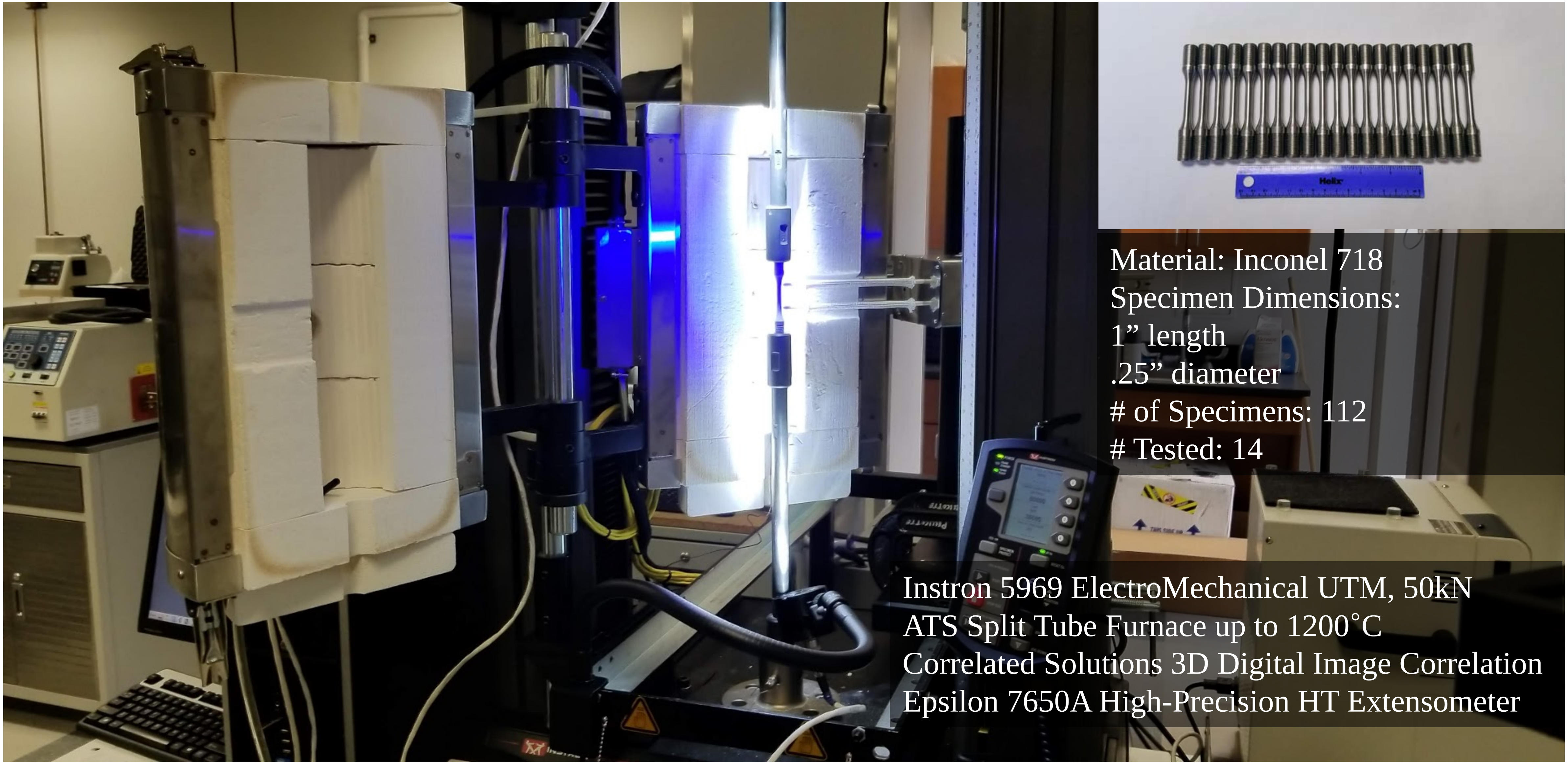
Dr. Jack Chessa
Associate Professor
Department of Mechanical Engineering
Co-Principal Investigator
jfchessa@utep.edu

Jacob T. Pellicotte
Undergraduate Research Assistant
jtpellicotte@miners.utep.edu

Conferences

- Conferences
  - Mach, R. Hynes, A. Pellicotte, J. And Stewart, C.M., 2019. “Assessment Of Long Term Creep Using Strain Rate Matching From The Stepped Isostress Method And Stepped Isothermal Method,” ASME TurboExpo 2019. Phoenix, Arizona, June 17-21 2019 (Paper Accepted)
  - Pellicotte, J. Cotto, M. and Stewart, C.M., 2019.“Assessment Of Calibration Approaches For The Stress Relaxation Test,” ASME TurboExpo 2019. Phoenix, Arizona, June 17-21 2019 (Paper Accepted)
  - Mach, R. Hynes, A. Pellicotte, J. And Stewart, C.M., 2019. “Application of High Temperature Digital Image Correlation and Scanning Electron Microscopy to Accelerated Creep Testing,” Southwest Emerging Technology Symposium, El Paso, Texas, March 26-27
- Journal Articles
  - Mach, R. Haynes, A. Pellicotte, J. And Stewart, C.M., 2019. “Accelerated Creep Testing of Metallic Materials using the Stepped Isostress Method (SSM),” *Engineering Failure Analysis Journal* (in preparation)
  - Pellicotte, J. Mach, R. Cotto, M. and Stewart, C.M., 2019.“Accelerated Creep Testing Program for Advanced Creep Resistant Alloys for High Temperature Fossil Energy Applications ,” *Materials at High Temperatures Journal* (in preparation)

Material and Equipment

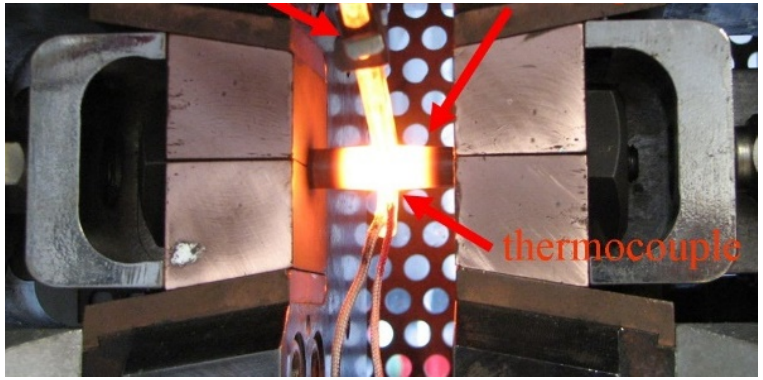


Material: Inconel 718
Specimen Dimensions:
1” length
.25” diameter
# of Specimens: 112
Tested: 14

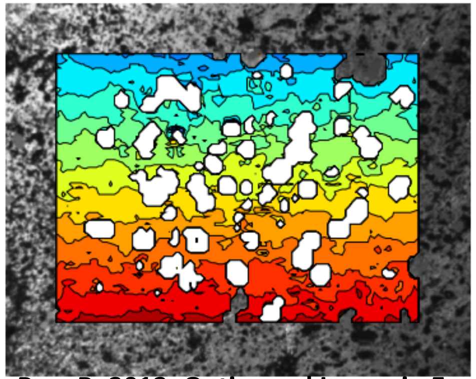
Instron 5969 ElectroMechanical UTM, 50kN
ATS Split Tube Furnace up to 1200°C
Correlated Solutions 3D Digital Image Correlation
Epsilon 7650A High-Precision HT Extensometer

Material and Equipment

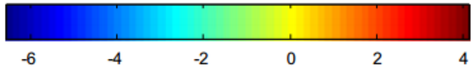
Incandescence at High Temperature



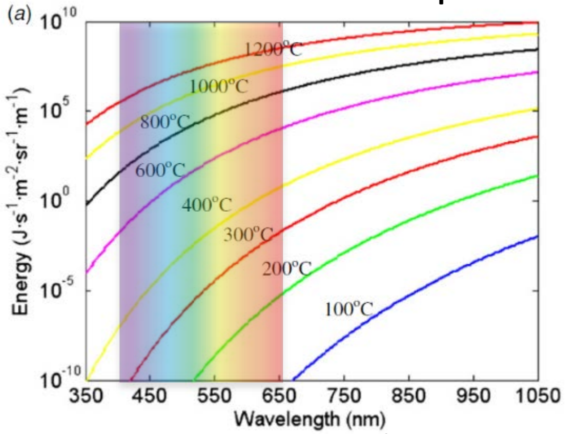
Gaps in correlation



Pan, B. 2012. Optics and Lasers in Eng.

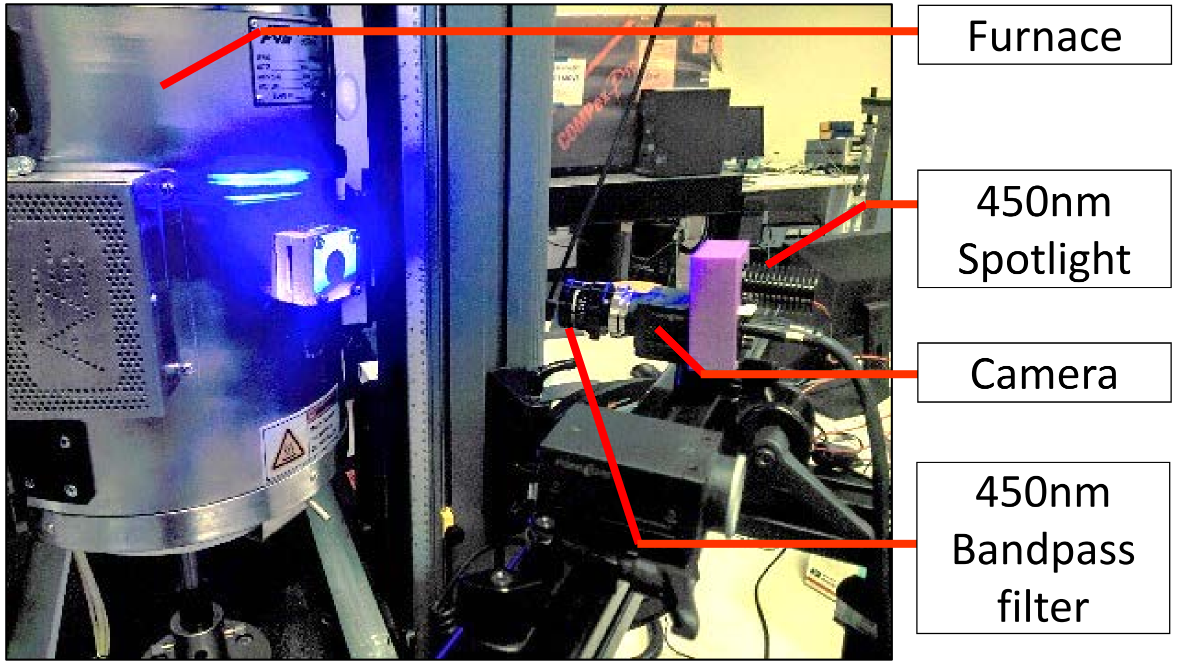


Correction Map

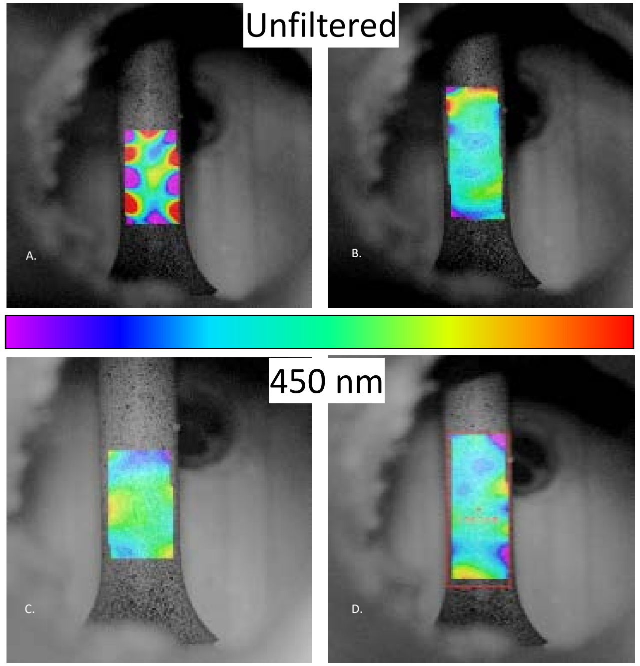


Pan, B. 2011. Beijing University of A&A.

MERG HT DIC System

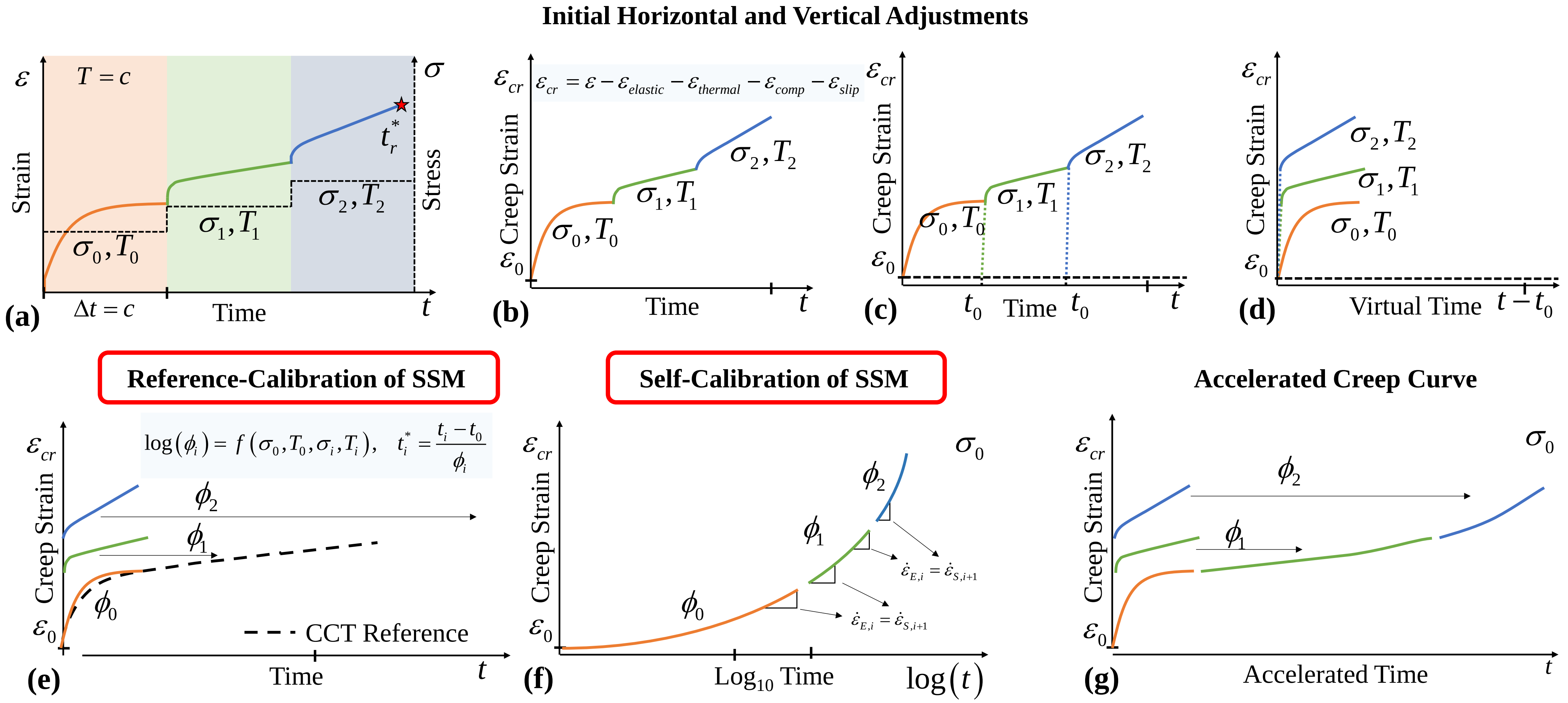


MERG Preliminary Data



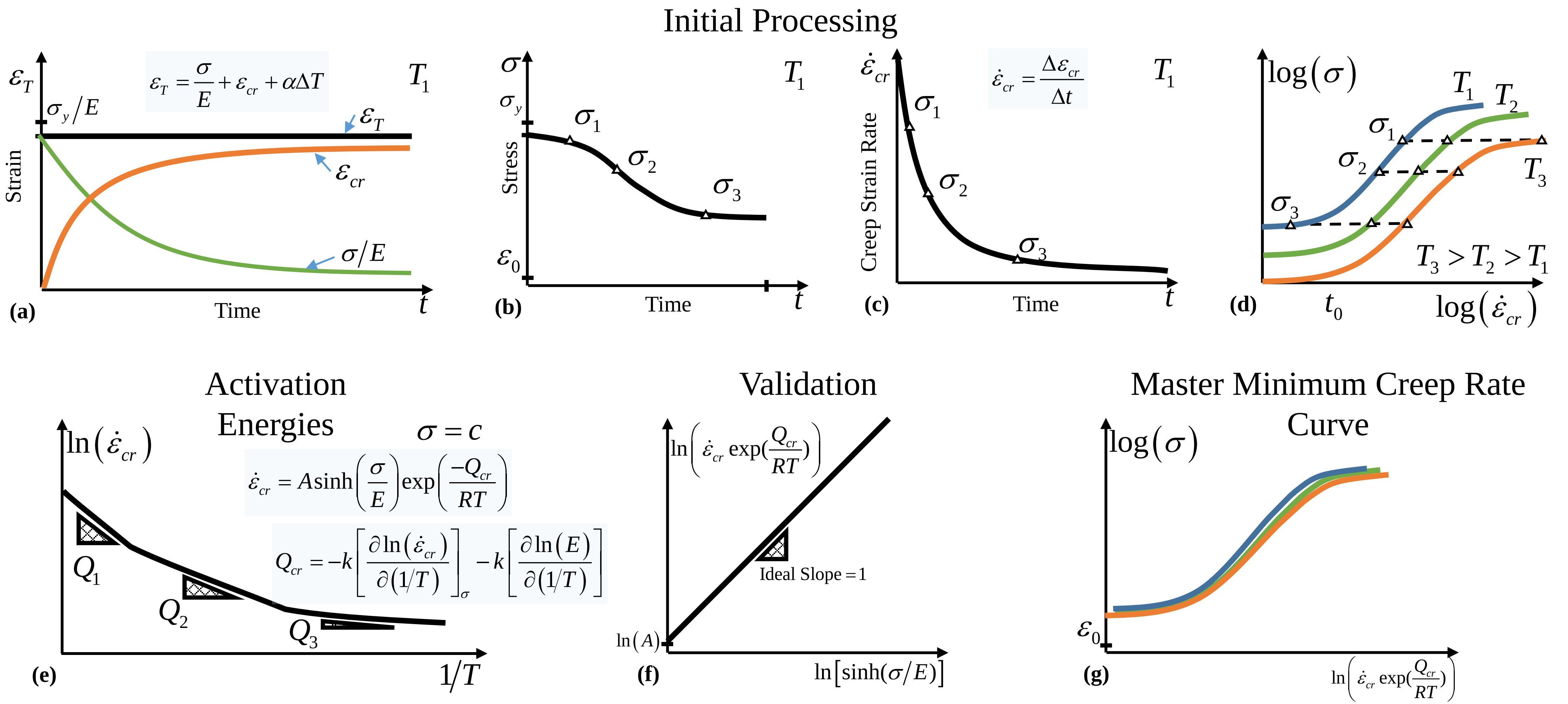
SSM Procedure

- SSM and SIM consist of holding stress or temperature for a set time before elevating the stress or temperature. Once the stress or temperature is elevated it is held again for the same time and the process repeats until failure.

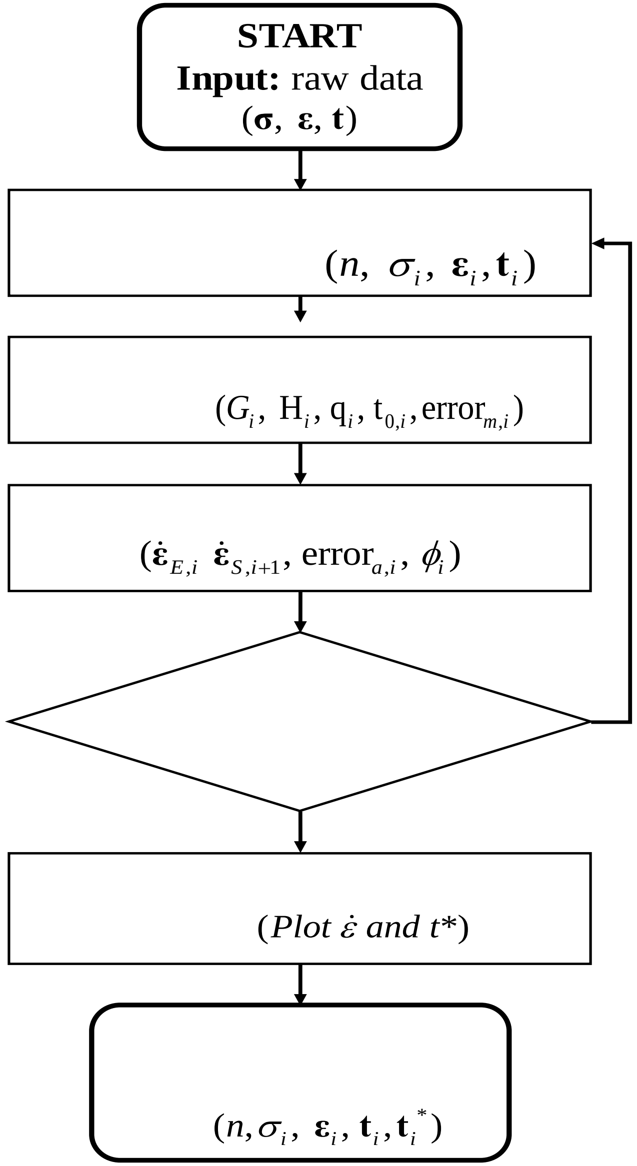


SRT Procedure

- A test matrix of Stress Relaxation Tests (SRTs) are performed to build multiple isotherms of minimum creep strain rate data, calculate creep activation energies, and develop a steady state creep deformation mechanism map.



SSM Software



- The code takes the data input from an excel and organizes it based on the number stress steps from an SSM test
- The virtual time starts are obtained using modified Theta-projection model:

$$\varepsilon = \theta_1 \left\{1 - \exp\left[-\theta_2 \left(t - t_0\right)\right]\right\} + \theta_3 \left\{\exp\left[\theta_4 \left(t - t_0\right)\right] - 1\right\}$$

- The Time shifts are calculated by using the following formula:

$$\phi_{i+1} = \frac{\phi_i \cdot \dot{\varepsilon}_{E,i}}{\dot{\varepsilon}_{S,i+1}}$$

- Accelerated Time is calculated as:

$$t^*_i = \frac{t_i - t_{0,i}}{\phi_i}$$

SSM/SIM Constraint Equation

Two relationships have been developed to determine the effect of stress on the rate of creep, (1) modified Williams-Landel-Ferry (WLF) equation and (1) Eyring equation

Williams-Landel-Ferry (WLF) Method (Jazouli, 2005) – Free volume theory

$$\dot{\varepsilon} = A\exp\left[B\left(\frac{1}{f}-1\right)\right]$$

$$f = f_0 + \alpha_T\left(T - T_0\right) + \alpha_\sigma\left(\sigma - \sigma_0\right)$$

$$\log\left(\phi_{\sigma T}\right) = -C_1\left[\frac{C_3\left(T-T_0\right)+C_2\left(\sigma-\sigma_0\right)}{C_2C_3+C_3\left(T-T_0\right)+C_2\left(\sigma-\sigma_0\right)}\right]$$

Assume Constant *T*

$$\log\left(\frac{\dot{\varepsilon}_1}{\dot{\varepsilon}_2}\right) = \frac{-C_1\left(\sigma_2-\sigma_1\right)}{C_3+\left(\sigma_2-\sigma_1\right)} \Rightarrow$$

Convert to iterative

$$\log\left(\phi_i\right) = \frac{-C_1\left(\sigma_i-\sigma_0\right)}{C_3+\left(\sigma_i-\sigma_0\right)}$$

Eyring Method (Giannopoulos,2011) – Creep Micromechanics, thermally activated plastic flow

$$\dot{\varepsilon} = \dot{\varepsilon}_0\exp\left(\frac{-Q^*}{RT}\right)\exp\left(\frac{V^*\sigma}{k_bT}\right)$$

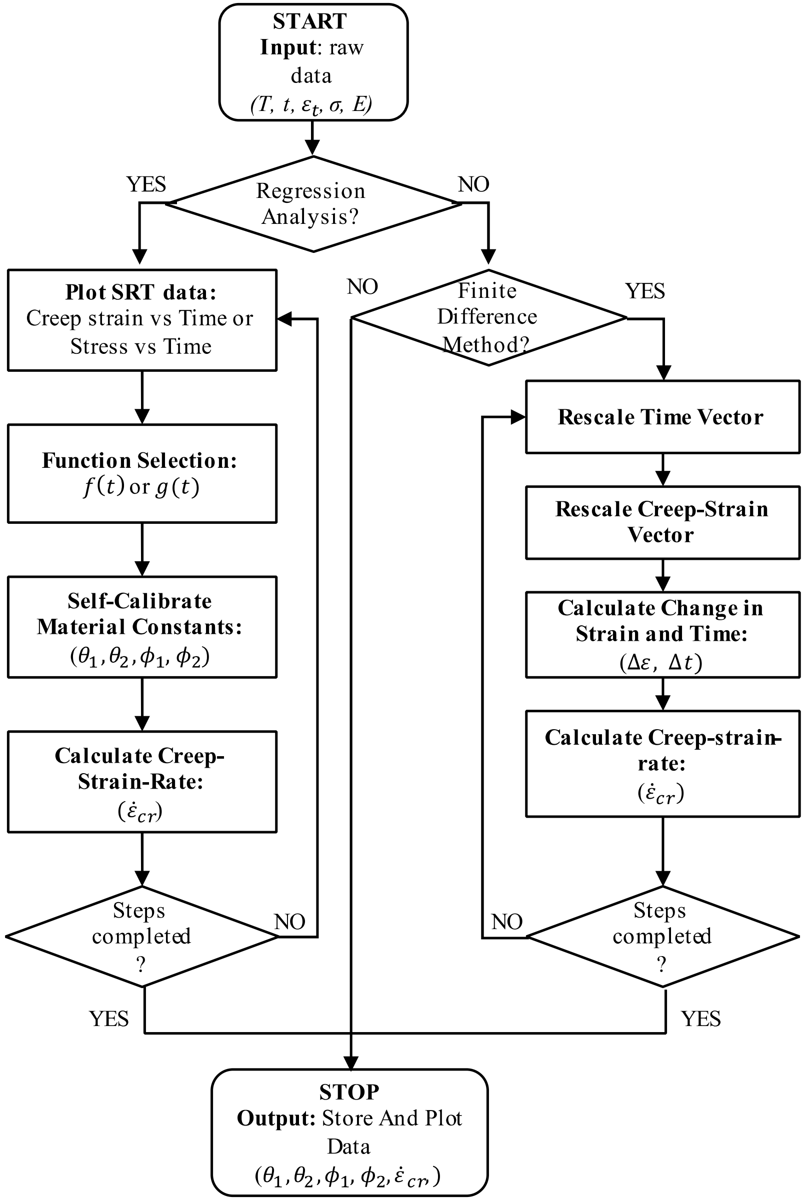
Assume Constant *T*

$$\ln\left(\frac{\dot{\varepsilon}_1}{\dot{\varepsilon}_2}\right) = \frac{V^*}{k_bT}\left(\sigma_1-\sigma_2\right) \Rightarrow$$

Convert to log and iterative

$$\log\left(\phi_i\right) = \frac{-V^*}{2.3k_bT}\left(\sigma_i-\sigma_0\right)$$

SRT Software



- The MATLAB code is divided into distinct categories, regression analysis and finite difference, which takes the raw data inputted from an excel file to produce plots:
  - Stress vs time
  - Creep strain vs time
  - Creep strain rate vs stress

- There are 3 calibration options that arise from the total strain

$$\varepsilon_{cr}(t) = \varepsilon_{tot} - \frac{\sigma(t)}{E} - \alpha\Delta T$$

  - Finite Difference of creep strain vs time (7)

$$\dot{\varepsilon}_{cr} = \frac{\Delta\varepsilon_{cr}}{\Delta t}$$

  - Regression analysis of stress vs time (4)

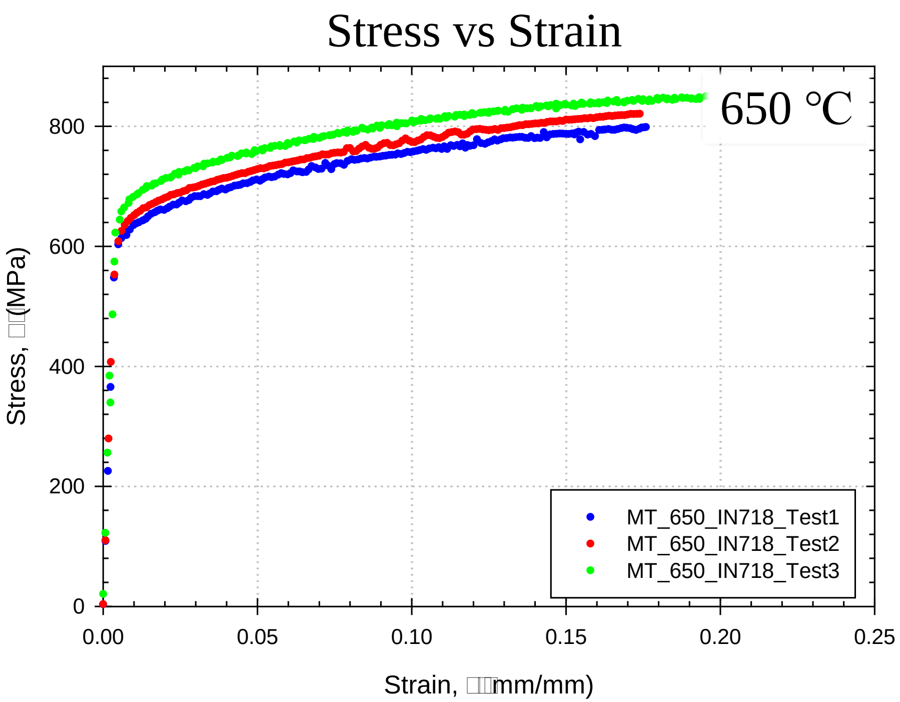
$$\dot{\varepsilon}_{cr} = -\frac{d}{dt}\left[\frac{f(t)}{E}\right] \qquad \text{where,} \quad f(t) = -\theta_1 \log(t) + \theta_2$$

  - Regression analysis of creep strain vs time (6)

$$\dot{\varepsilon}_{cr} = \frac{d}{dt}[g(t)] \qquad \text{where,} \quad g(t) = \phi_1 \log(t) + \phi_2$$

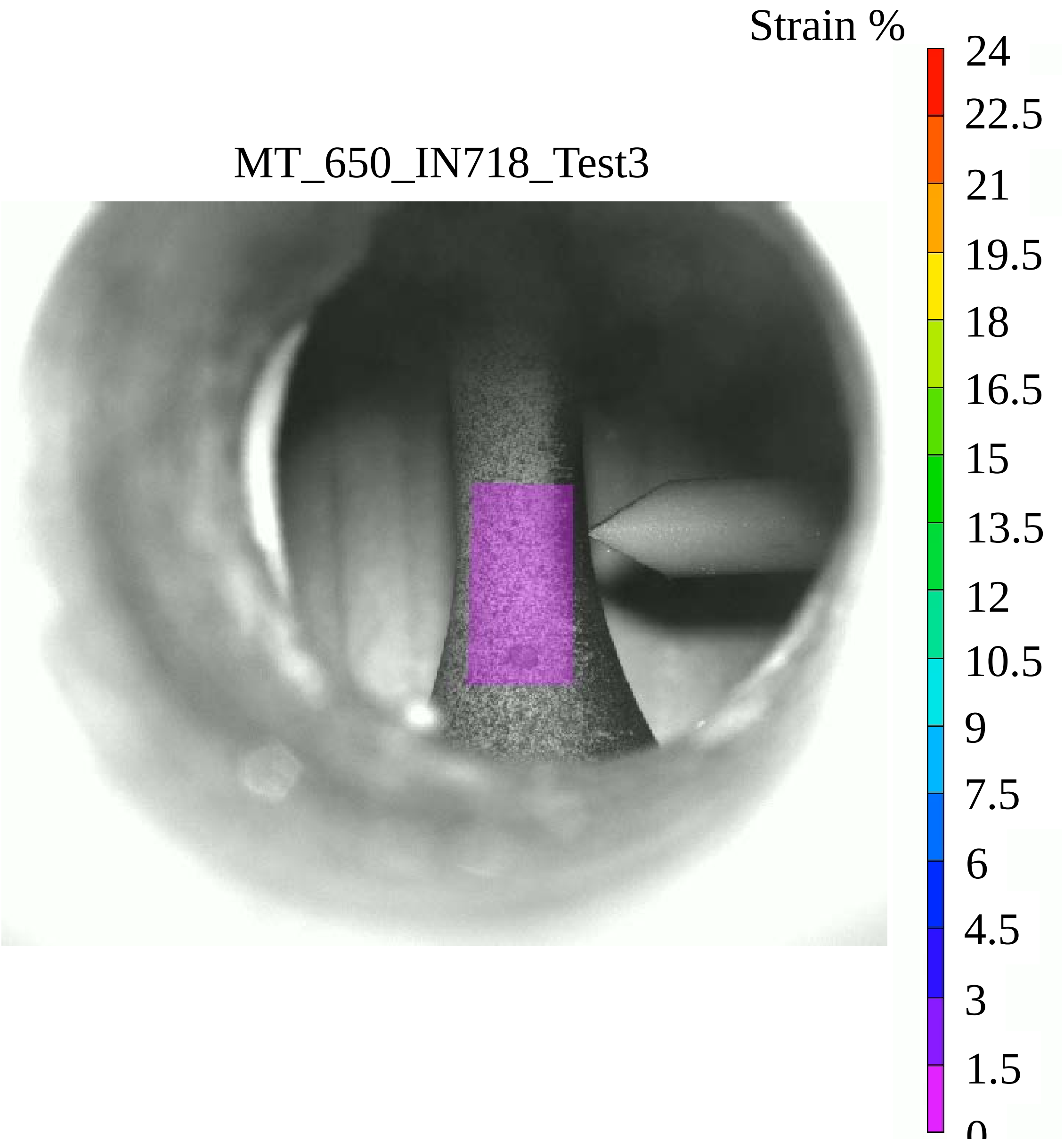
Experimental Results

- Monotonic Tensile Tests (MTs) were performed to collect modulus, yield strength, and ultimate tensile strength.
- This data was used for
  - defining the boundary conditions for the proposed CCTs and ACTs
  - verifying the testing machine
  - and determining how the procured material compare to the legacy experimental database

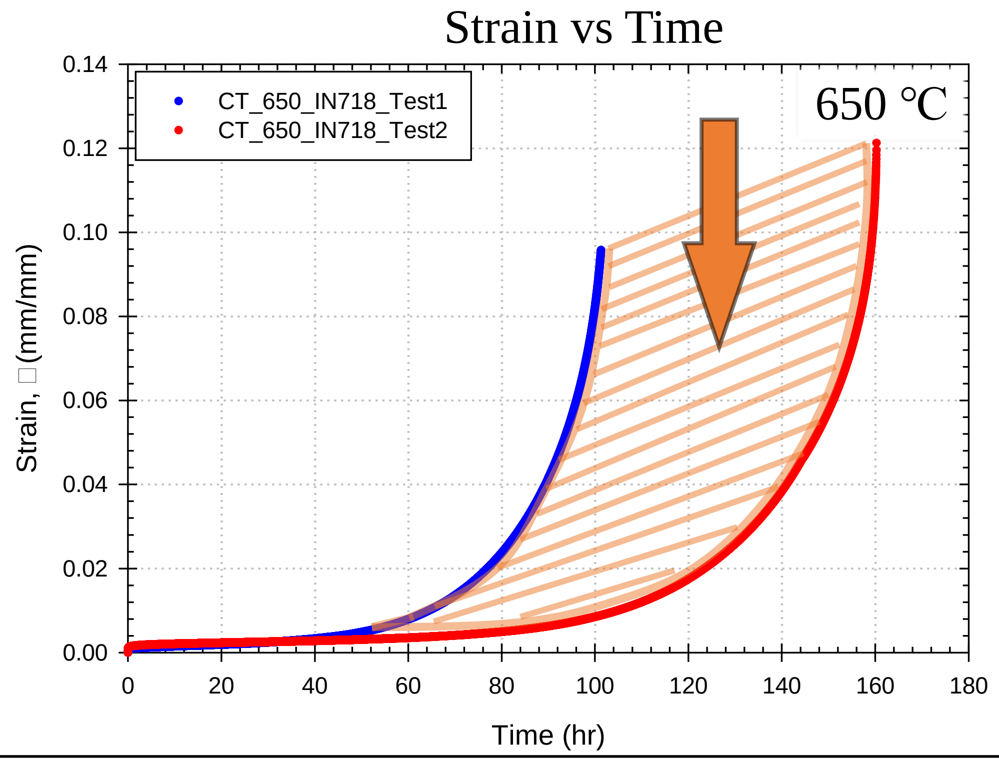


Specimen ID	UTS (MPa)	YS (MPa)	Young’s Modulus (GPa)
MT_IN718_650_Test1	798.6	613.6	157.1
MT_IN718_650_Test2	820.6	625.3	153.4
MT_IN718_650_Test3	854.1	664.3	147.5
Average	824.4	634.4	152.7
Coefficient of Variance	3.38	4.18	3.17

2D Digital Image Correlation



Experimental Results



- The short-term conventional creep tests (CCTs) were preformed to produce creep data.
- Tests were designed to not exceed 168 hours.
- The short-term creep data is used to act as the high-resolution data needed to be quantitively compared to the ACTs to determine the overall quality of the calibration approaches.

Specimen ID	Stress (MPa)	Temperature (°C)	Rupture Time (hr)	Final Creep Strain %	Minimum-Creep-Strain Rate	Adjusted Elastic and Plastic Creep Strain (mm/mm)
CT_650_IN718_Test1	636.0	650	101.3	10.0	3E-05	0.0042
CT_650_IN718_Test2	636.0	650	160.3	12.7	5E-05	0.0061
Coefficient of Variance	-	-	31.89	16.82	35.35	25.49

SRT Test Matrix Design

- The objective of these stress relaxation tests is to determine the extent that prior SRT testing has on subsequent tests.
- By comparing the consistency of the SRT results at a single isotherm, we will validate whether it is reasonable to gather multiple isotherms of SRT data using a single specimen.

Step 1- Calculate the theoretical constant strain

$$\varepsilon_{total} = \frac{0.9 * \sigma_{ys}}{E} \qquad \varepsilon_{total\_rate} = \frac{\varepsilon_{total}}{60}$$

where,

σ_{ys} is the *Yield Strength* of the specimen

E is the material's *Modulus of Elasticity*

Step 2- Calculate the theoretical displacement control needed for constant strain.

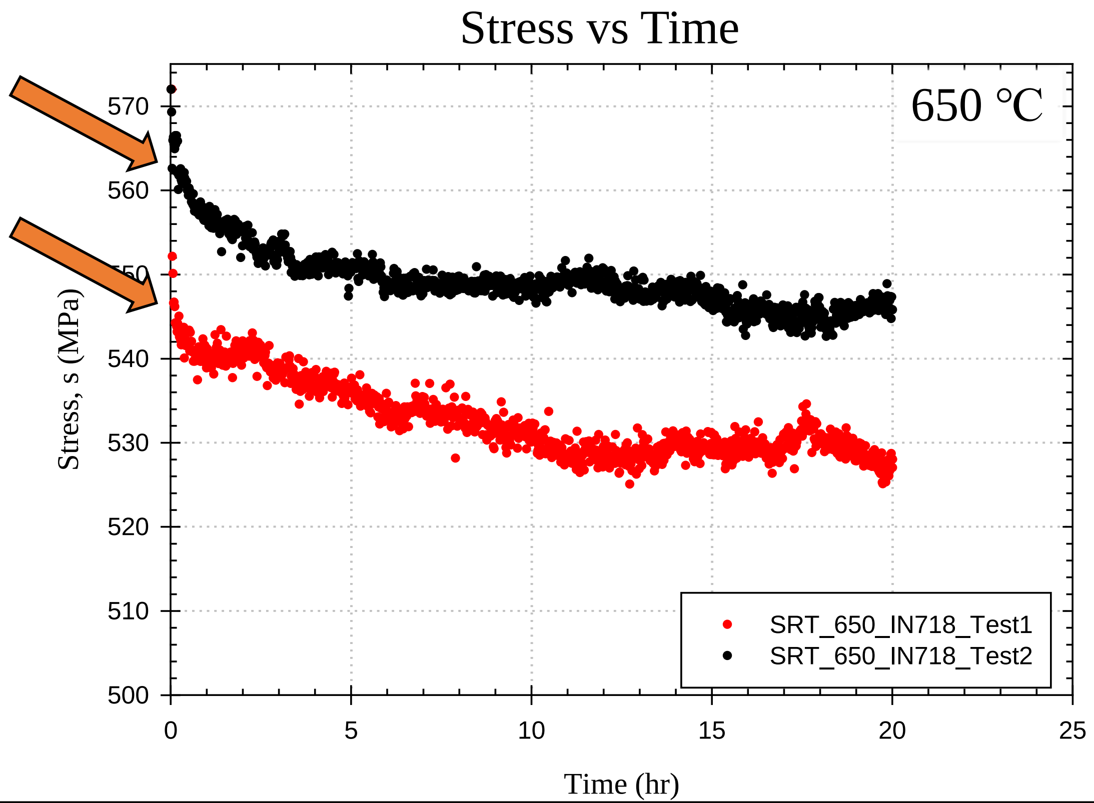
$$\delta = \varepsilon_{total} * L \qquad \delta_{rate} = \frac{\delta}{60\,\text{sec}}$$

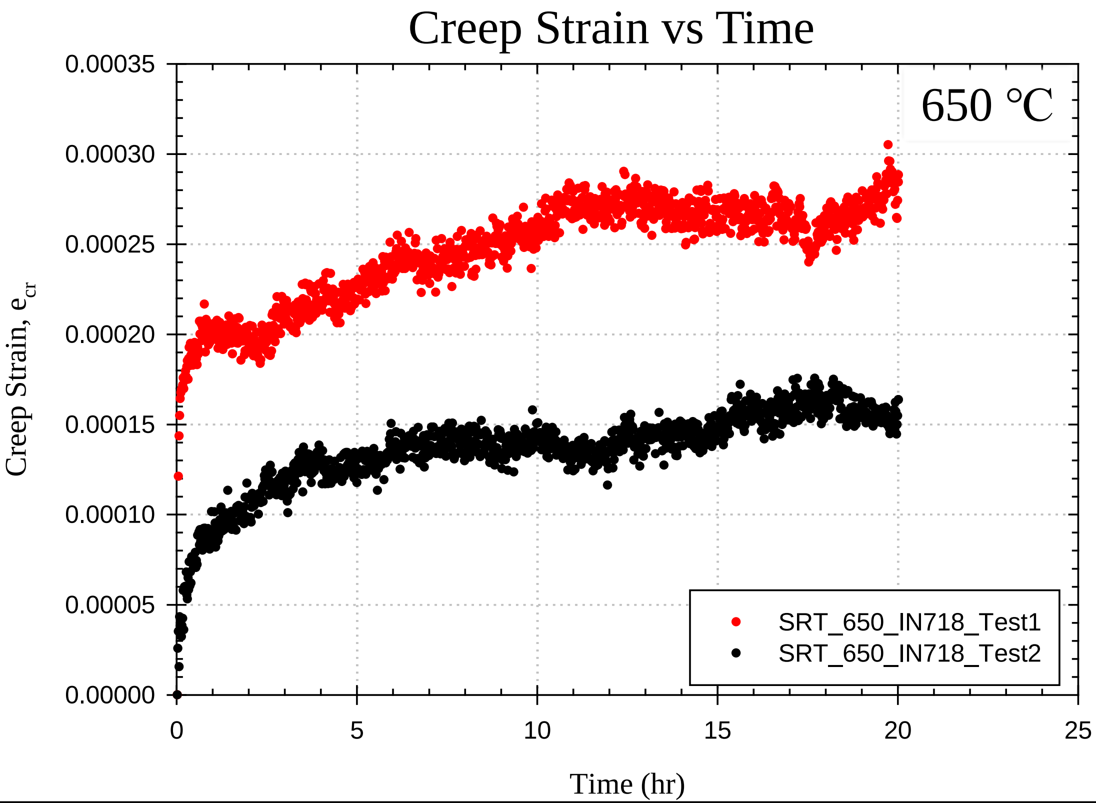
where,

L is the *Gage Length* of the specimen

Specimen ID	Temperature (°C)	Total Time (hr)	Initial Stress (MPa)	Total Strain (mm/mm)	Total Strain Rate (1/s)	Displacement (mm)	Displacement Rate (mm/s)
SRT_650_IN718 _Test	650	20	572	.0037	6.2 E-06	.095	.0016

SRT Data

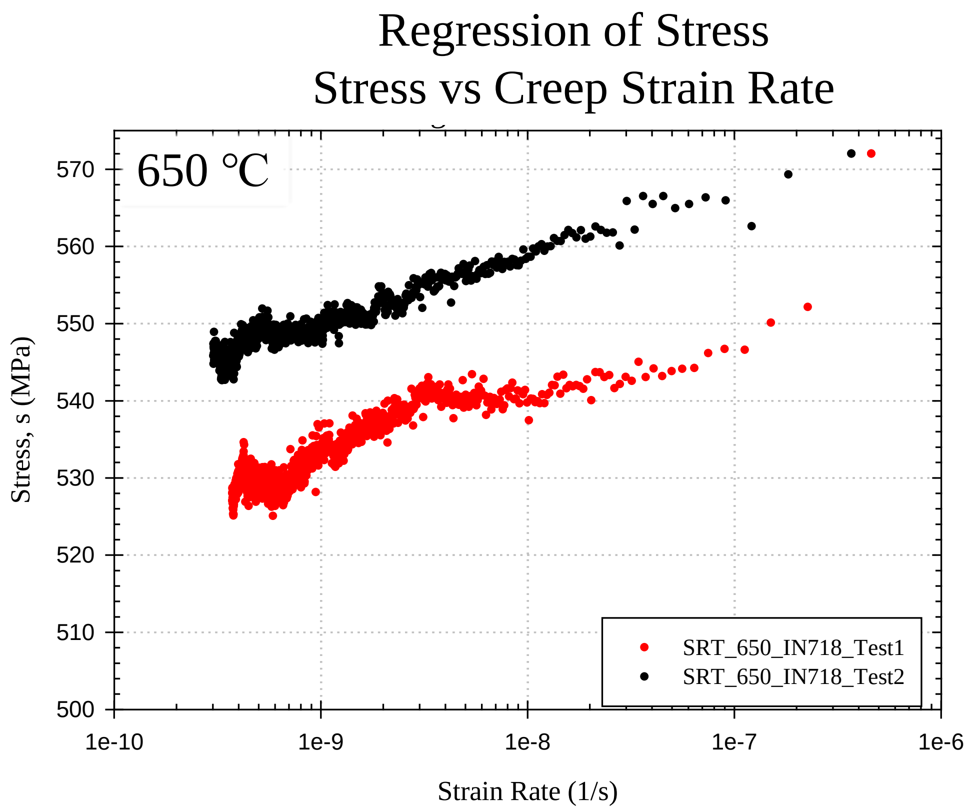


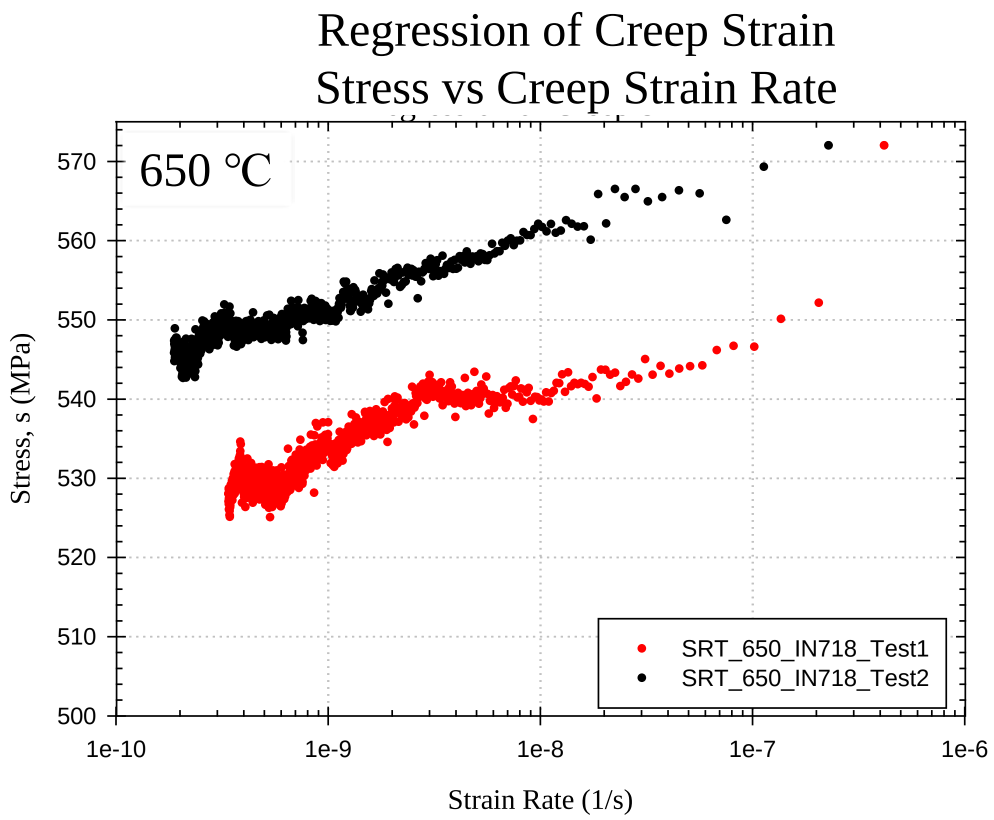


Specimen ID	Temperature (°C)	Total Time (hr)	Initial Stress (MPa)	Final Stress (MPa)	Total Strain (mm)
SRT_650_IN718_Test1	650	20	572	528.0	.00349
SRT_650_IN718_Test2	650	20	572	545.8	.00366
Coefficient of Variance	-	-	-	2.3	2.6

SRT Results

- The SRT results indicate that repeated SRTs on a single specimen are not feasible as there are small amounts of accumulation of creep between tests
- This suggests that it is not feasible to gather multi-isotherms using SRT which will guide in the decision making process for other test matrices.





SRT Optical Microscopy

- Tests were conducted in the Elastic Regime resulting in an absence of noticeable deformation
- Oxidation

Image taken from painted area

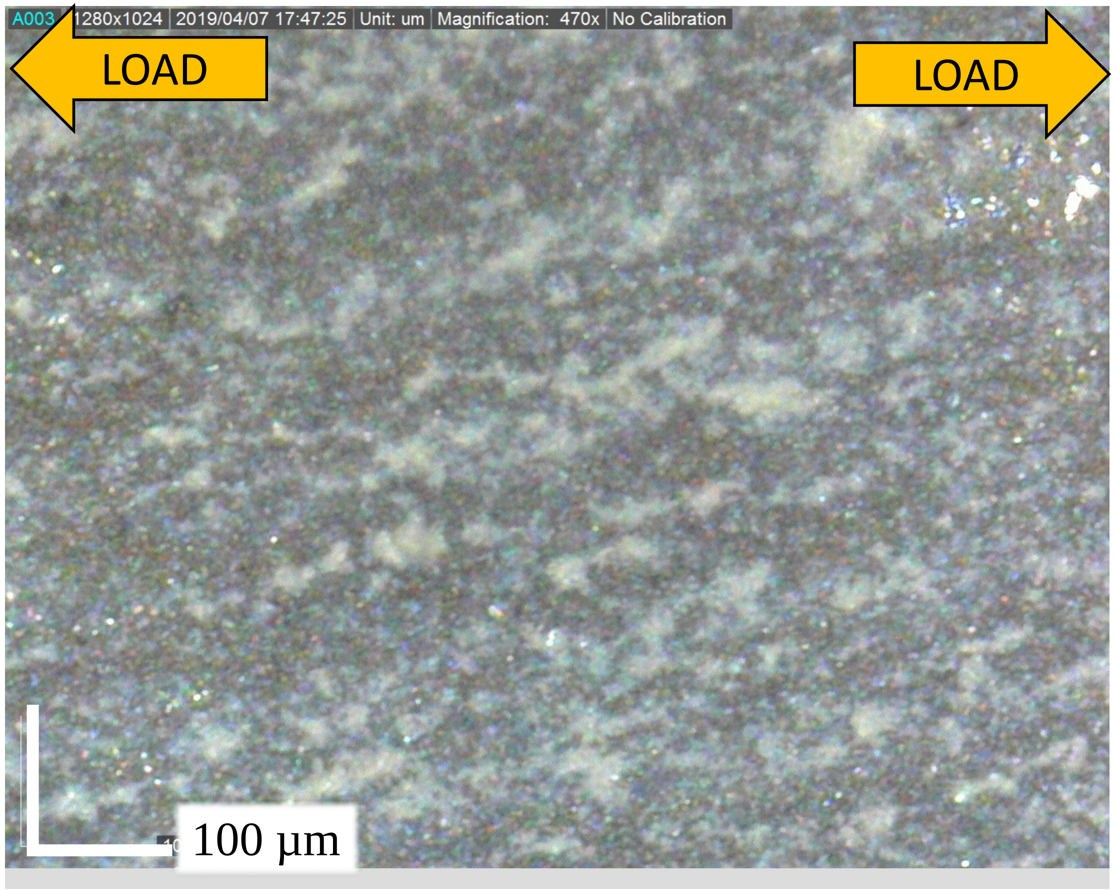
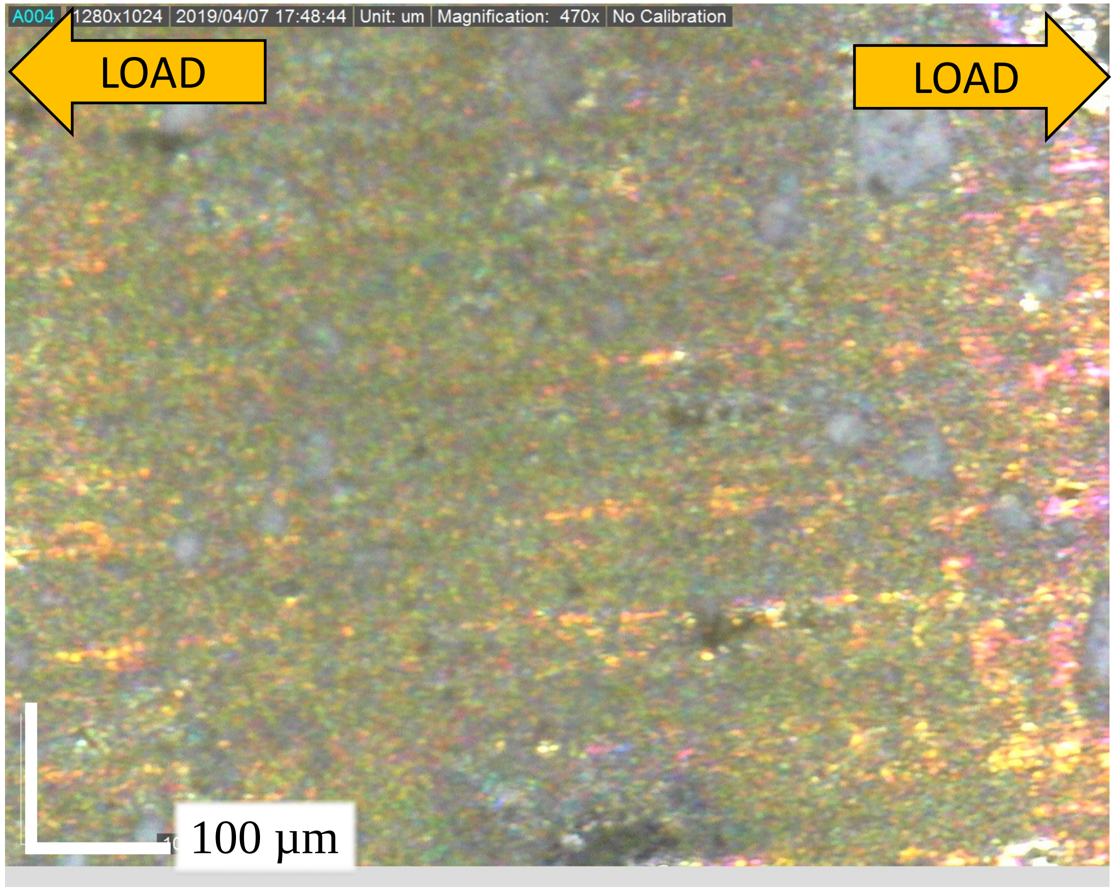


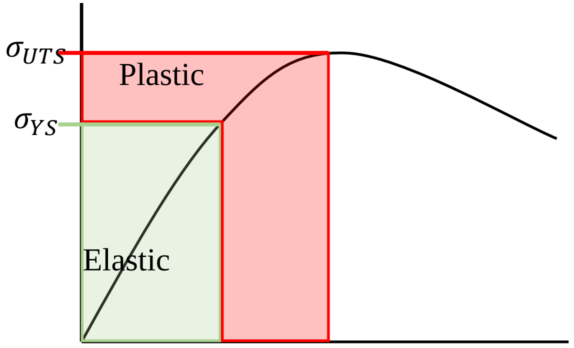
Image taken from non painted area



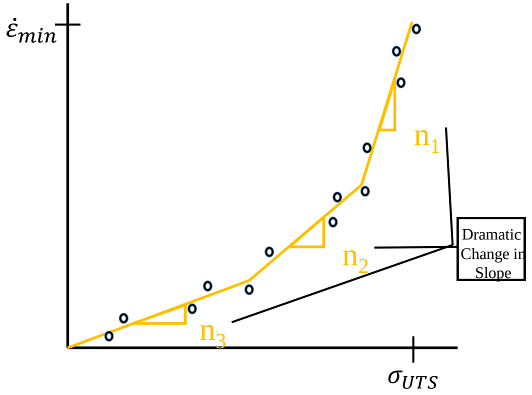
SSM Test Matrix Design

Step 1 – Mechanism Transition

Identify the deformation mechanisms in the region of interest so that the initial and final stress may be selected. It is preferred to avoid mechanism transitions during an experiment (if possible).



Monotonic Tension



MCR

Step 2 – Stress-Step Magnitude

Calculate the stress increment based on the number of steps desired.

$$\Delta\sigma = \frac{\sigma_N - \sigma_0}{N - 1}$$

Step 3 – Estimate SSM Test Duration

Calibrate a rupture prediction model to existing conventional creep test data (if available). Herein, the preferred model is the Sin-Hyperbolic rupture equation.

$$t_r(\sigma) = \frac{1}{M \sinh(\frac{\sigma}{\sigma_{ys}})^{\chi}}$$

Apply Miner's rule to estimate the real-time duration of an SSM test needed to rupture the specimen.

$$\sum_{i=1}^{N-1} \frac{\Delta t}{t_r(\sigma_i)} + \frac{\Delta t + \Delta t^*}{t_r(\sigma_N)} = 1 \qquad t_{total} = N \cdot \Delta t + \Delta t^*$$

Test-Parameter Decision Matrix

Test-parameter decision matrix for SIM and SSM

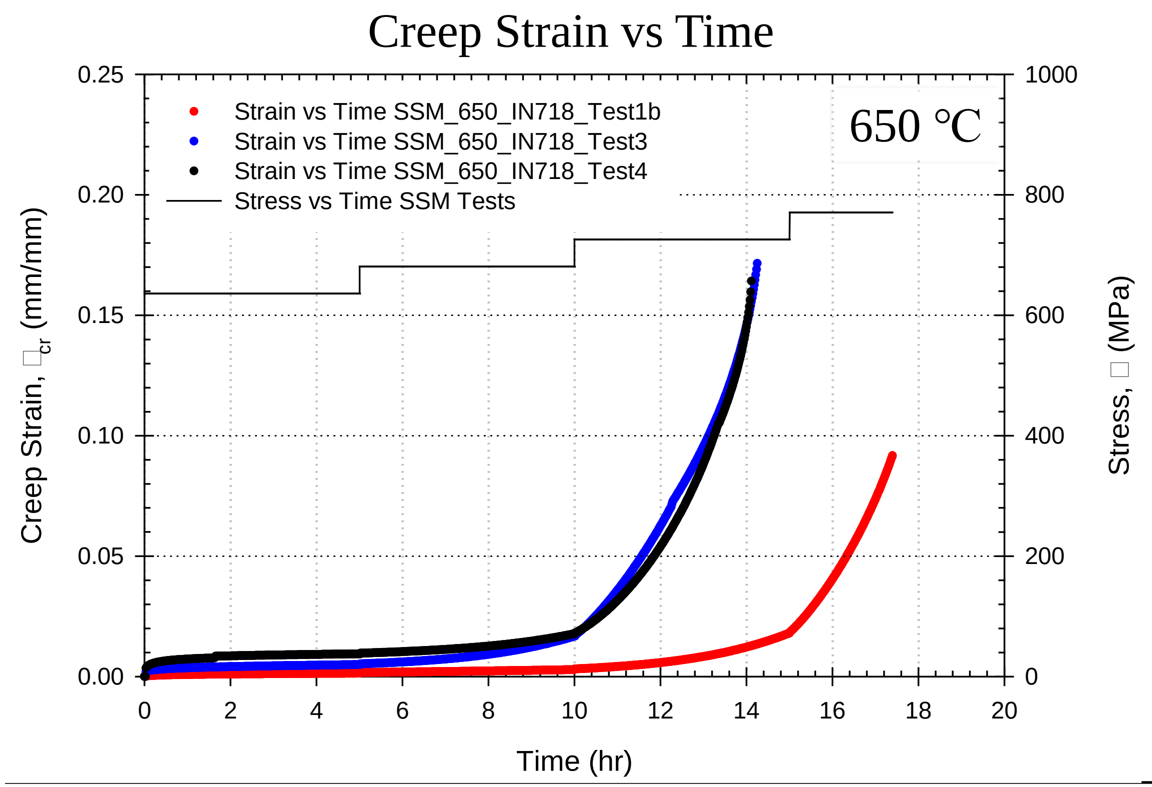
SIM	Challenges	Maximize Acceleration?
σ_0	If $\sigma_0 \to 0$ the load cell and/or extensometer may not be able to record creep.	σ_0 set to the design stress.
T_0	If $T_0 \to 0$ is too small, extensometer may not be able to record creep.	T_0 set to the design temperature.
ΔT_0	If $\Delta T_0 \leftarrow 0$, the T_i steps may not be visible in extensometer data and could be below the error of the temperature probe.	$\Delta T \to (T_T - T_0)/3$ where T_T is the temperature of the next mechanism transition.
Δt	If $\Delta t \to 0$ the creep curves will not capture the secondary creep regime needed for calibration of SIM.	$\Delta t \to 0$ minimizes real time thus maximizes the acceleration.
SSM	**Challenges**	**Maximize Acceleration?**
σ_0	If $\sigma_0 \to 0$ the load cell and/or extensometer may not be able to record creep.	σ_0 set to the design stress.
$\Delta\sigma_0$	If $\Delta\sigma_0 \leftarrow 0$, the σ_i steps may not be visible in extensometer data and could be below the error of the load cell.	$\Delta T \to (\sigma_T - T_0)/3$ where σ_T is the stress of the next mechanism transition.
T_0	If $T_0 \to 0$ is too small, extensometer may not be able to record creep.	T_0 set to the design temperature.
Δt	If $\Delta t \to 0$ the creep curves will not capture the secondary creep regime needed for calibration of SSM.	$\Delta t \to 0$ minimizes real time thus maximizes the acceleration.

SSM Test Matrix

- The purpose of this proof-of-concept SSM test matrix is to determine the hold times necessary to reach the minimum creep strain rate and stress increases for each step
- The following test matrix was designed to match the data gathered from short-term conventional creep test mentioned previously

Specimen ID	Stress (MPa)	Unaccelerated Rupture Time (hr)	Time Hold (hr)	Total Duration (Days)
SSM_650_IN718_Test	636	168	5	1.54
	681	96.8	5	
	726	55.9	5	
	771	32.4	21.86	

SSM Data

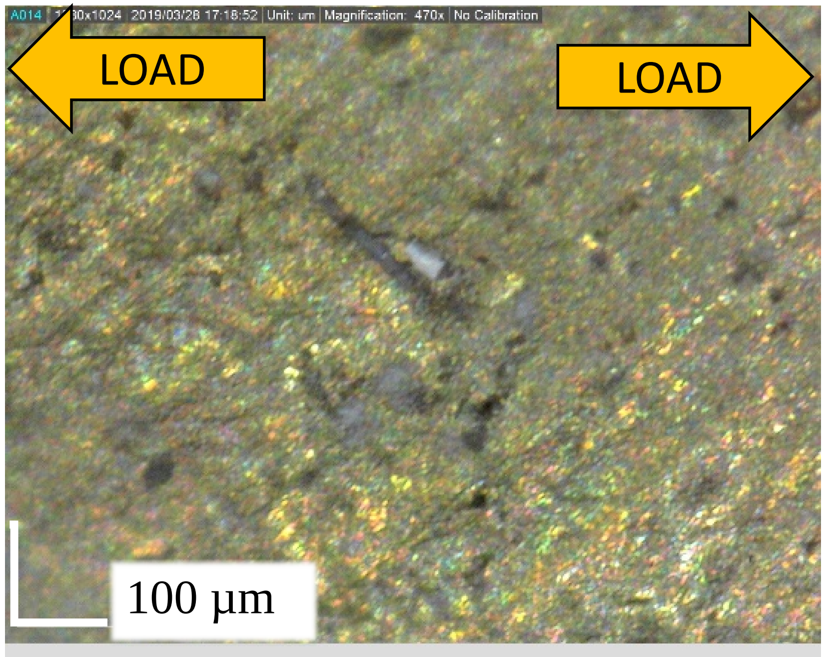


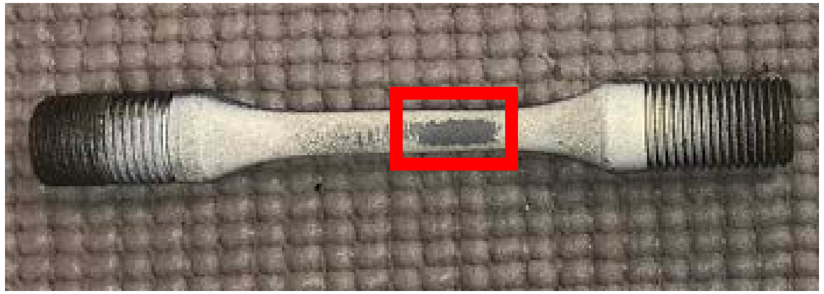
- These SSM tests were conducted within the plastic regime of IN718
- The coefficient of variance for the rupture between the 3 SSM experiments is smaller when compared to the 2 CT experiments, however, the coefficient of variance for the final creep strain for the SSM experiments is smaller than the CT experiments

Specimen ID	Testing Temperature (°C)	Rupture Time (hr)	Final Stress (MPa)	Final Creep Strain %
SSM_650_IN718_Test1b	650	17.4	771	10.0
SSM_650_IN718_Test3	650	14.3	725	17.2
SSM_650_IN718_Test4	650	14.1	725	16.4
Coefficient of Variance	-	12.11	3.58	27.15

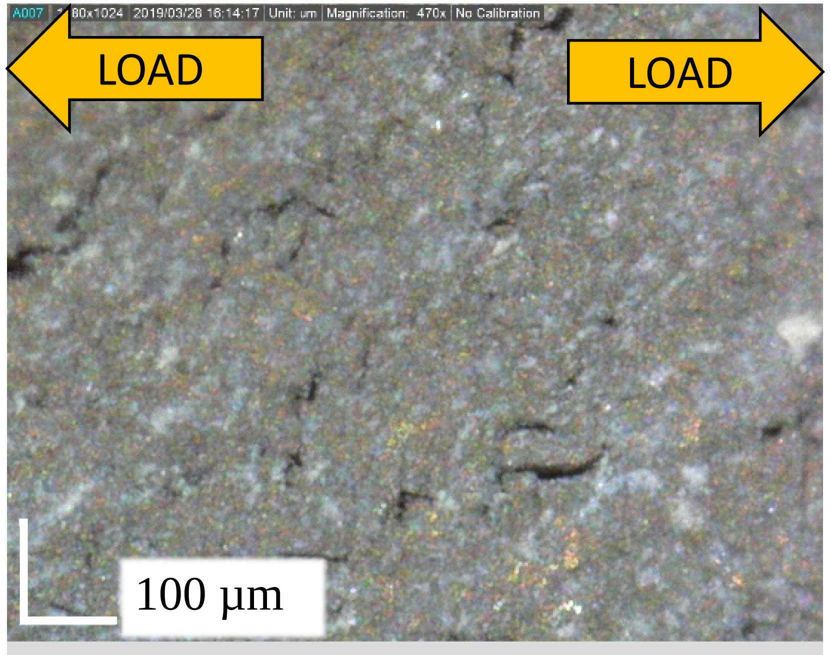
SSM Optical Microscopy

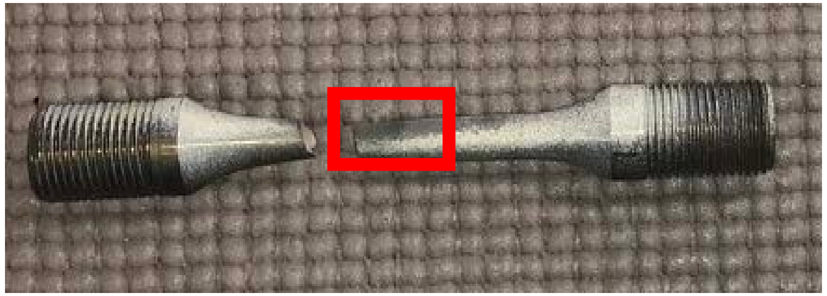
SSM_650_IN718_Test1b



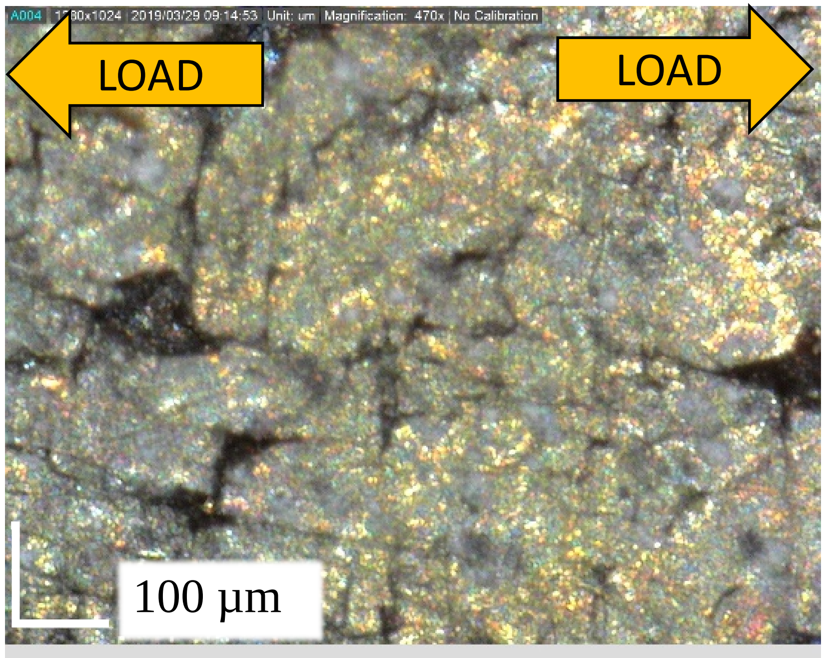


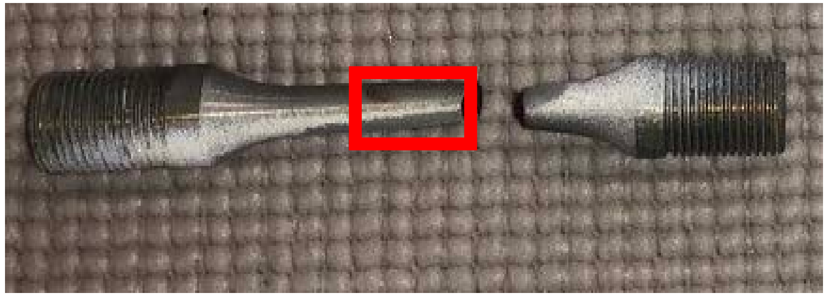
SSM_650_IN718_Test3



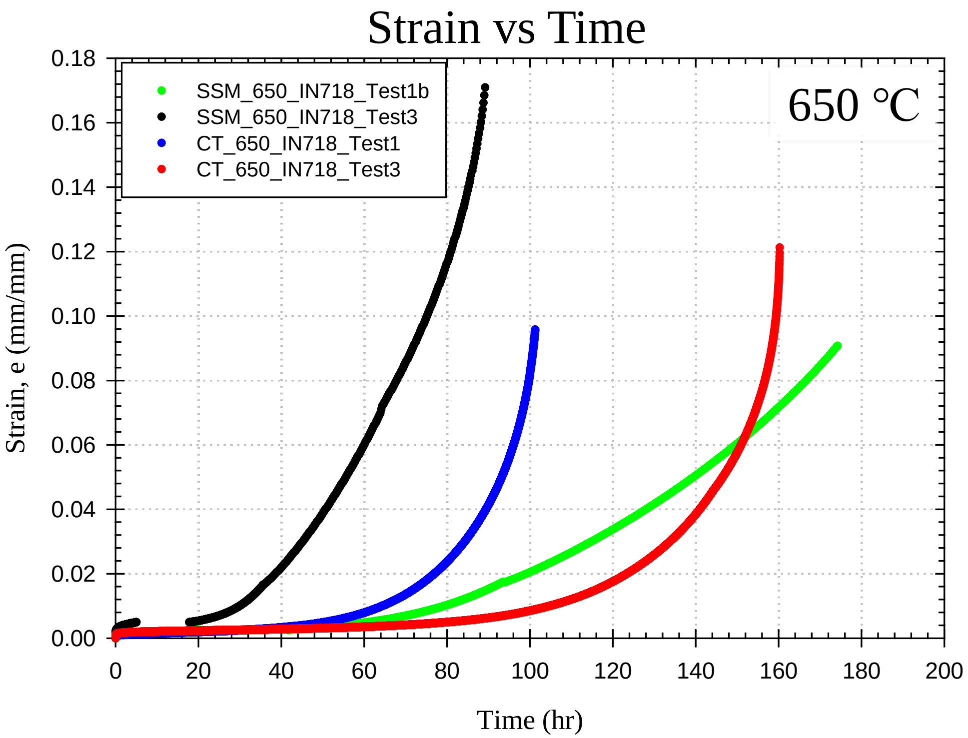


SSM_650_IN718_Test4





SSM Predictions

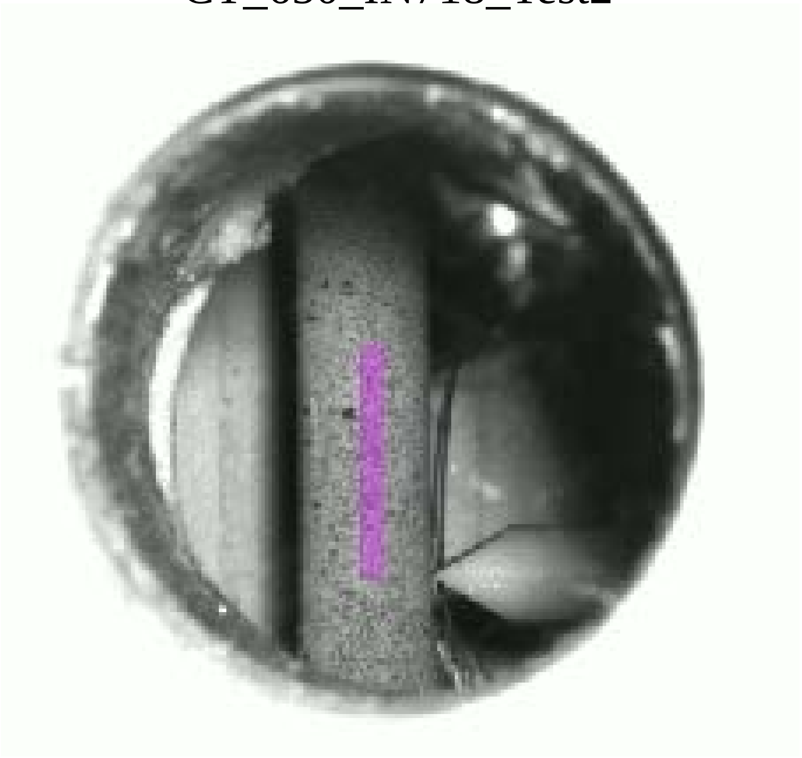


- A modified theta-project model was employed to determine the t_0 values. It was determined that a wide range of t_0 values can be used to produce credible creep curves. This created a problem post-calibration.
- During calibration, three stress constraints were considered, no constraint, WLF, and Eyring equation. Eyring equation produced the most consistent and smooth accelerated creep curves.
- Post-calibration, some portions of the accelerated creep curve did not align properly. The t_0 at these portions needed to be manually adjusted and calibration repeated in order to achieve a smooth accelerated creep curve. This second level optimization will be automated in the future.

Specimen ID	Stress (MPa)	Temperature (°C)	Rupture Time (hr)	Final Strain %	$t_0 2$	$t_0 3$	$t_0 4$	ϕ_1	ϕ_2	ϕ_3	ϕ_4
SSM_650_IN718_Test1b	636.0	650	174.2	9.1	3.7	6.0	12.2	1	.31	.09	.02
CT_650_IN718_Test1	636.0	650	101.3	10.0	-	-	-	-	-	-	-
CT_650_IN718_Test2	636.0	650	160.3	12.7	-	-	-	-	-	-	-

3D Digital Image Correlation

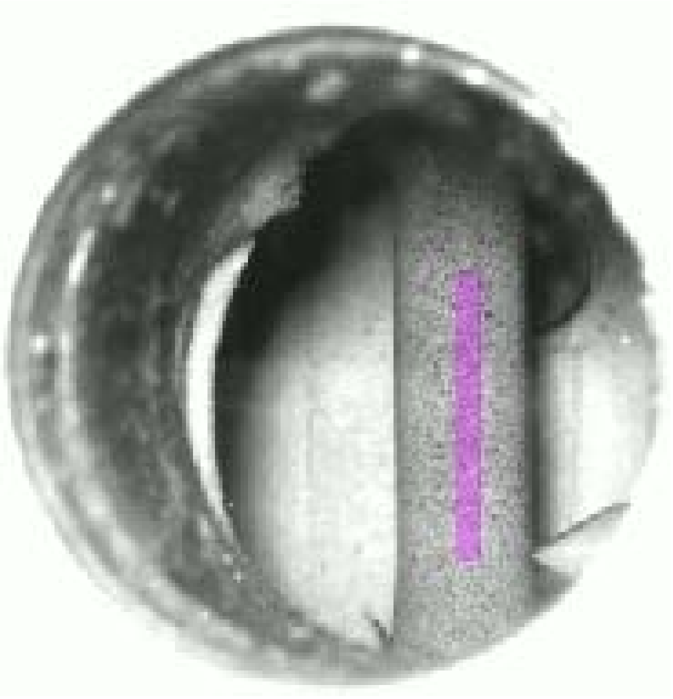
CT_650_IN718_Test2



Strain %

10
9.375
8.75
8.125
7.5
6.875
6.25
5.625
5
4.375
3.75
3.125
2.5
1.875
1.25
0.625
0

SSM_650_IN718_Test3



Strain %

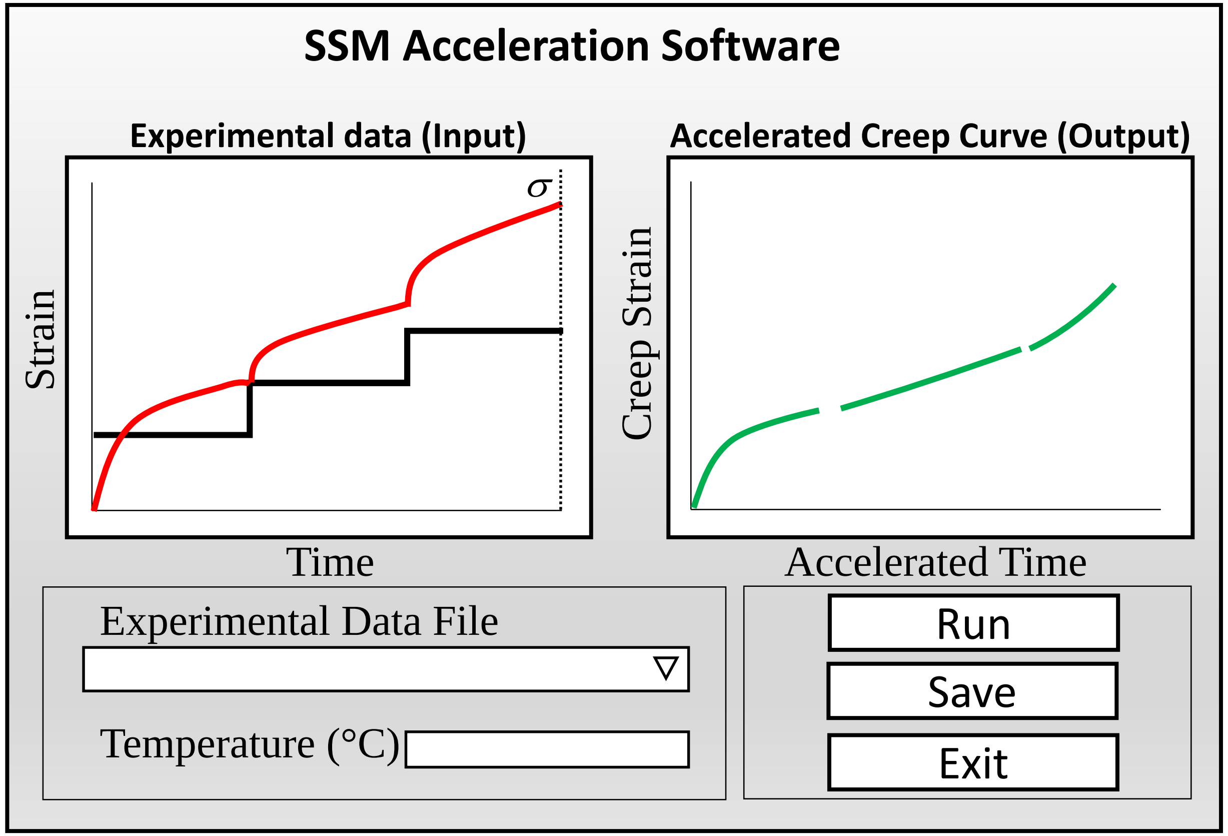
14.3
13.4
12.5
11.6
10.7
9.8
8.9
8.04
716
6.26
5.35
4.46
3.57
2.68
1.79
0.89
0

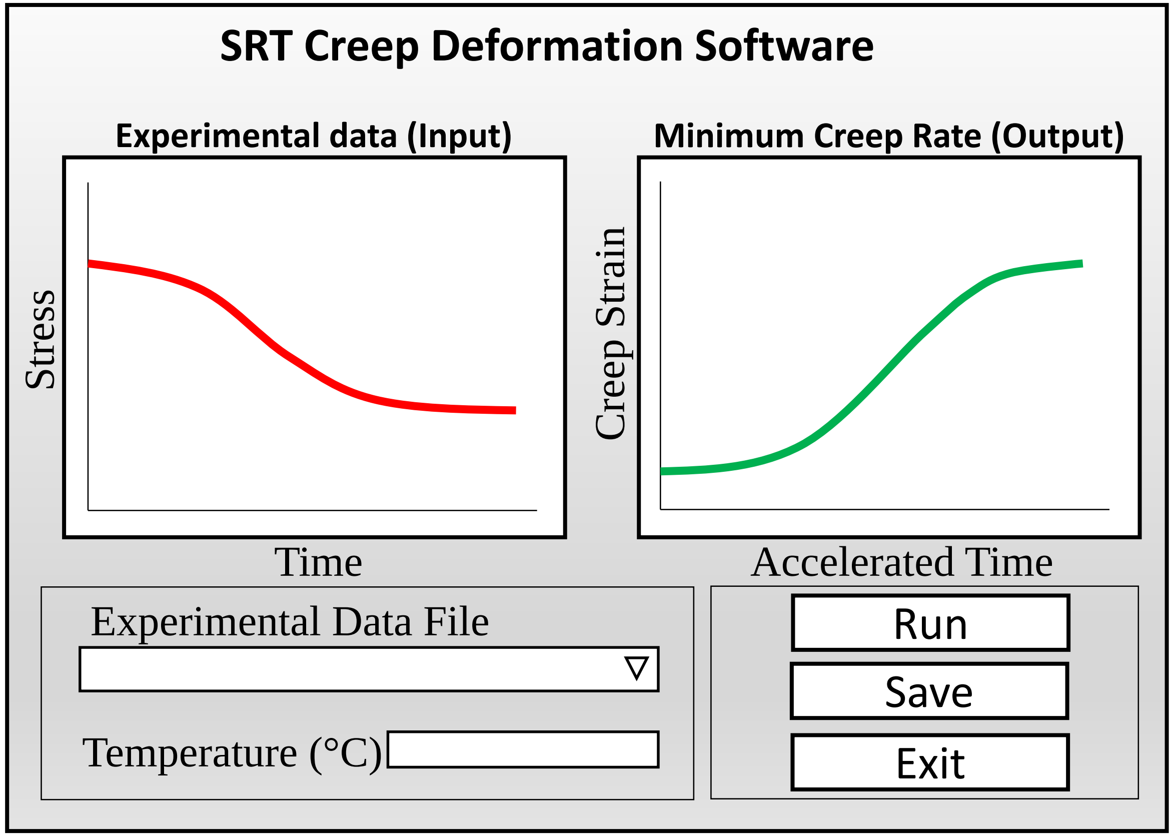
Next Steps for Goals/Objectives

- Continue to update ACT procedures
  - Conduct Stress Relaxation Tests on multiple specimens to obtain minimum creep strain rate data for IN718 at 650 °C
  - Optimize SSM test matrix to conduct tests at various stresses in the elastic and plastic regime of IN718
- Improve MATLAB software to include graphical user interface
- Post-Audit Validation of ACTs to reference data

Technology-To-Market

- **ASTM test standard**.
- Graphic user interfaces will be created allowing **FE material scientists to potentially reduce the time of implementation of new creep resistant alloys** from decades to months.





Conclusions

- As research and development continues for materials to operate within the new coal-fired power plants for 2030, there is a need to gather the creep deformation and creep rupture properties quickly.

- Results seen from our ACTs indicate that the SSM and SRT experiments are a feasible replacement to conventional creep testing; however, the challenges rely on further development of softwares, test matrices, and theory development.

THE UNIVERSITY OF TEXAS AT EL PASO

COLLEGE OF ENGINEERING

Robert M. Mach
Masters Research Assistant
rmmach@miners.utep.edu

Jacob T. Pellicotte
Undergraduate Research Assistant
jtpellicotte@miners.utep.edu

Calvin M. Stewart
Associate Professor of Mechanical Engineering
Director of the Materials at Extremes Research Group
cmstewart@utep.edu

MERG
UTEP MATERIALS AT EXTREMES RESEARCH GROUP

This material is based upon work supported by the Department of Energy National Energy Technology Laboratory under Award Number(s) DE-FOA-0001715.