

# **Creep Deformation and the Evolution of Residual Stresses in SOFC Anodes**

**Edgar Lara-Curzio, Andres Marquez, Ercan Cakmak, Beth Armstrong**  
Materials Science & Technology Division  
Oak Ridge National Laboratory

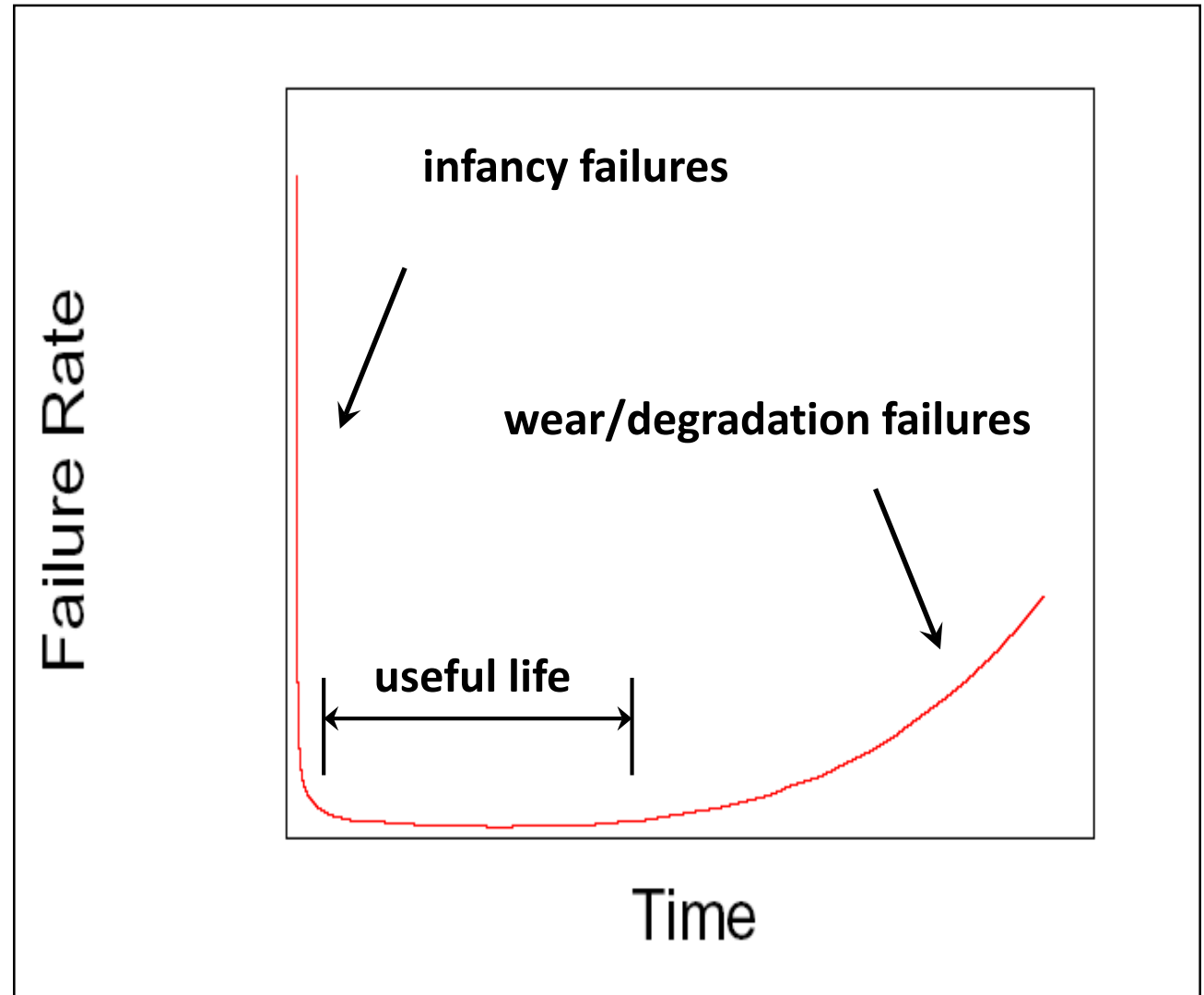
**Solid Oxide Fuel Cell (SOFC) Project Review Meeting**  
**Pittsburgh, PA June 13, 2017**

# Acknowledgments

This work was sponsored by the US Department of Energy, Office of Fossil Energy, Solid Oxide Fuel Cells Program, Core Technology Program at ORNL. We appreciate guidance and support from NETL program managers Rin Burke and Shailesh Vora.

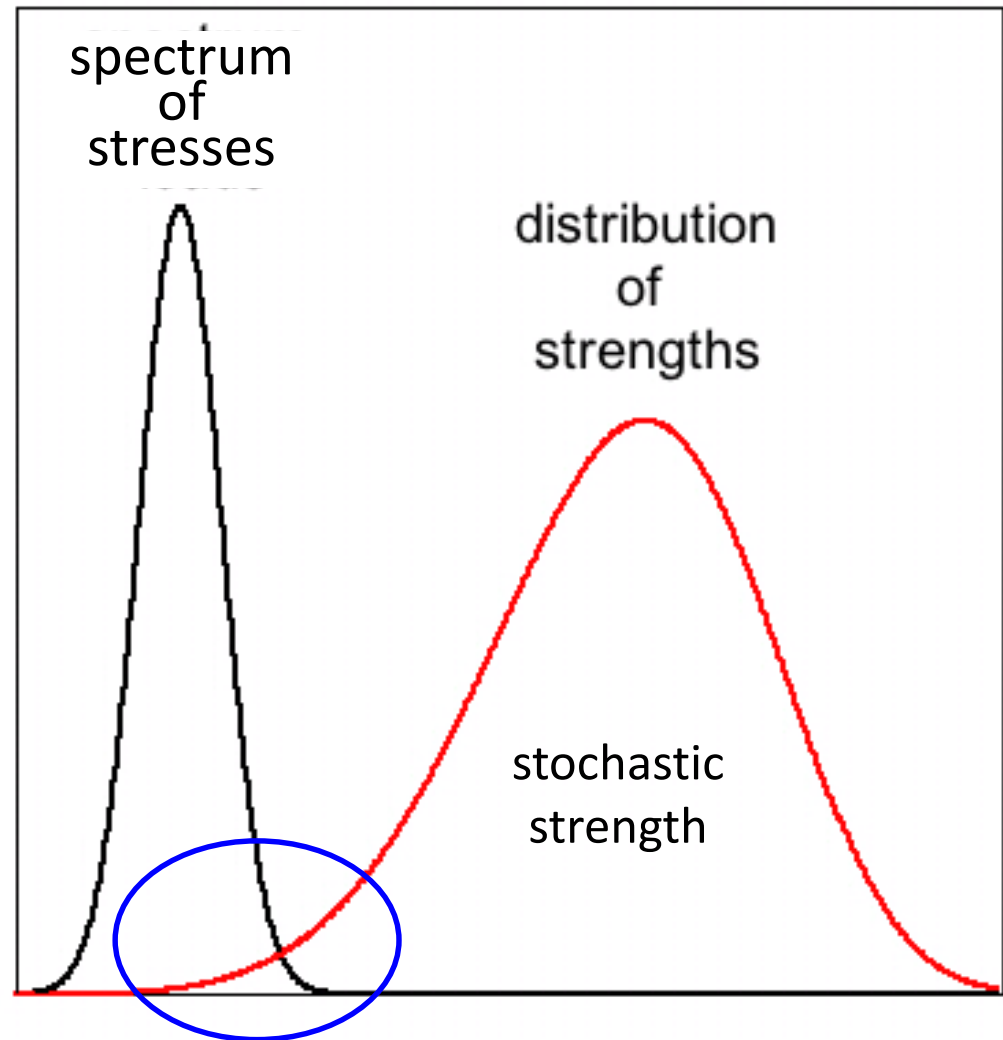
# Failure, Reliability and Durability of Systems

The failure rate of complex systems can be described by the bathtub curve

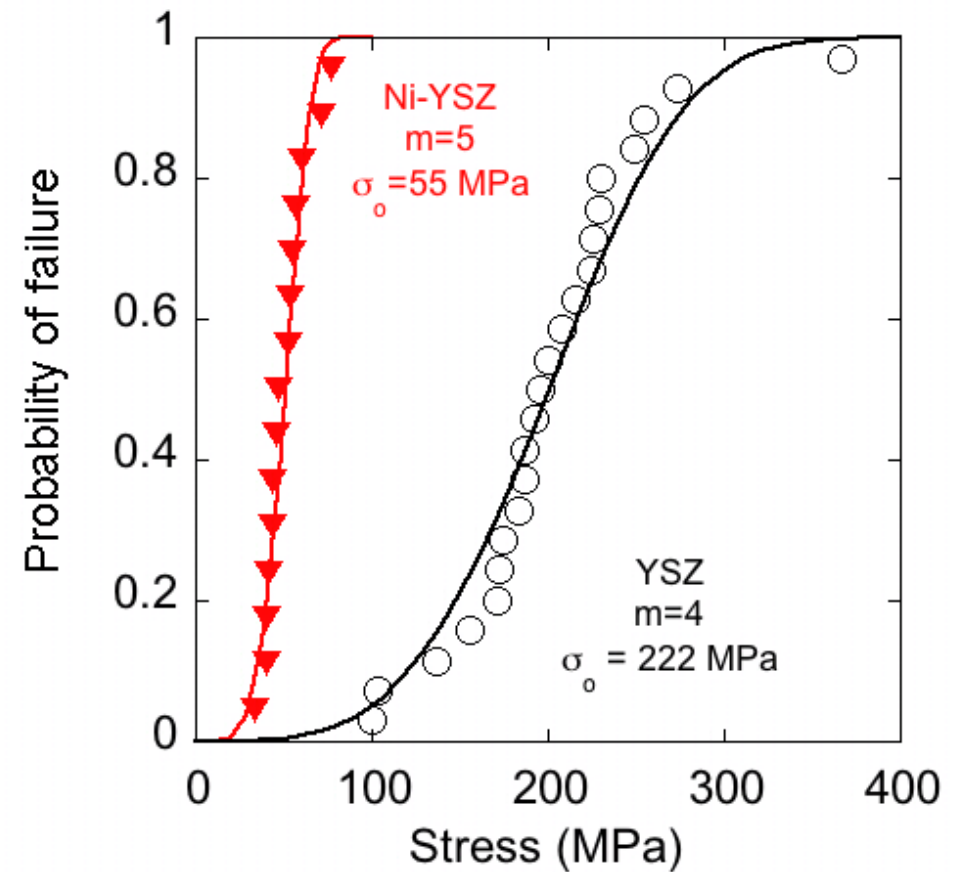
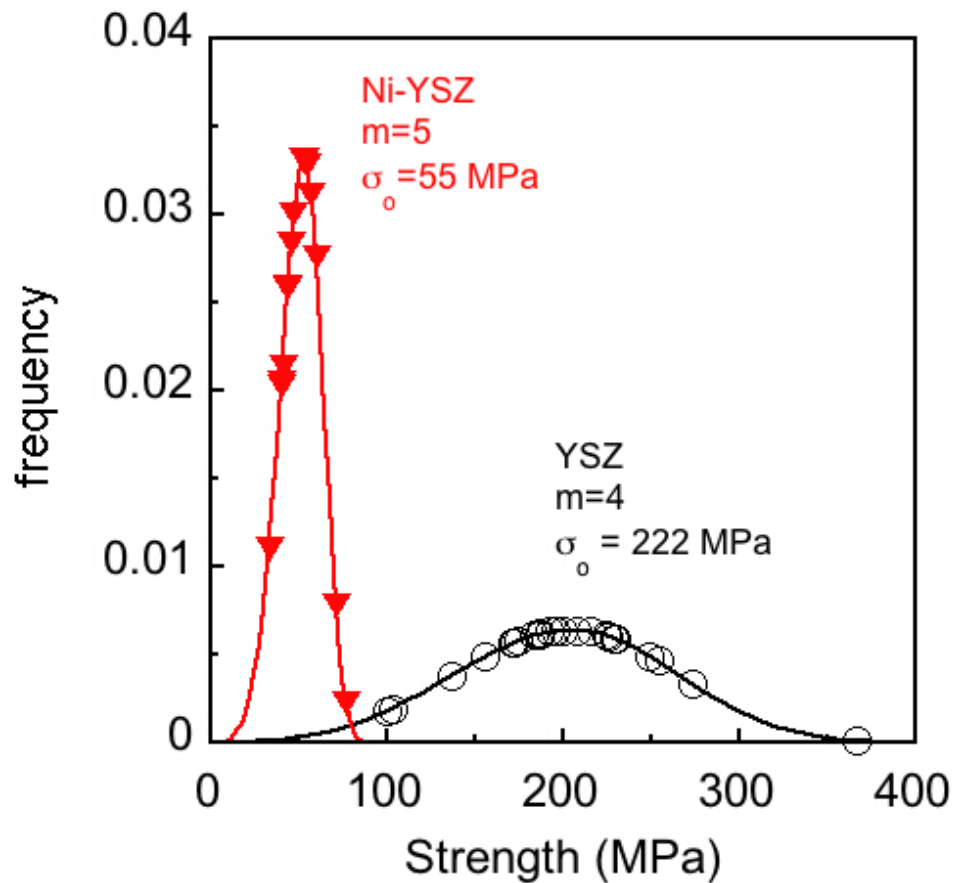


# Failure, Reliability and Durability of Systems

- Failure is determined by the intersection of the distributions of loads and strengths.
- The weakest elements of the population determine the reliability of the system.

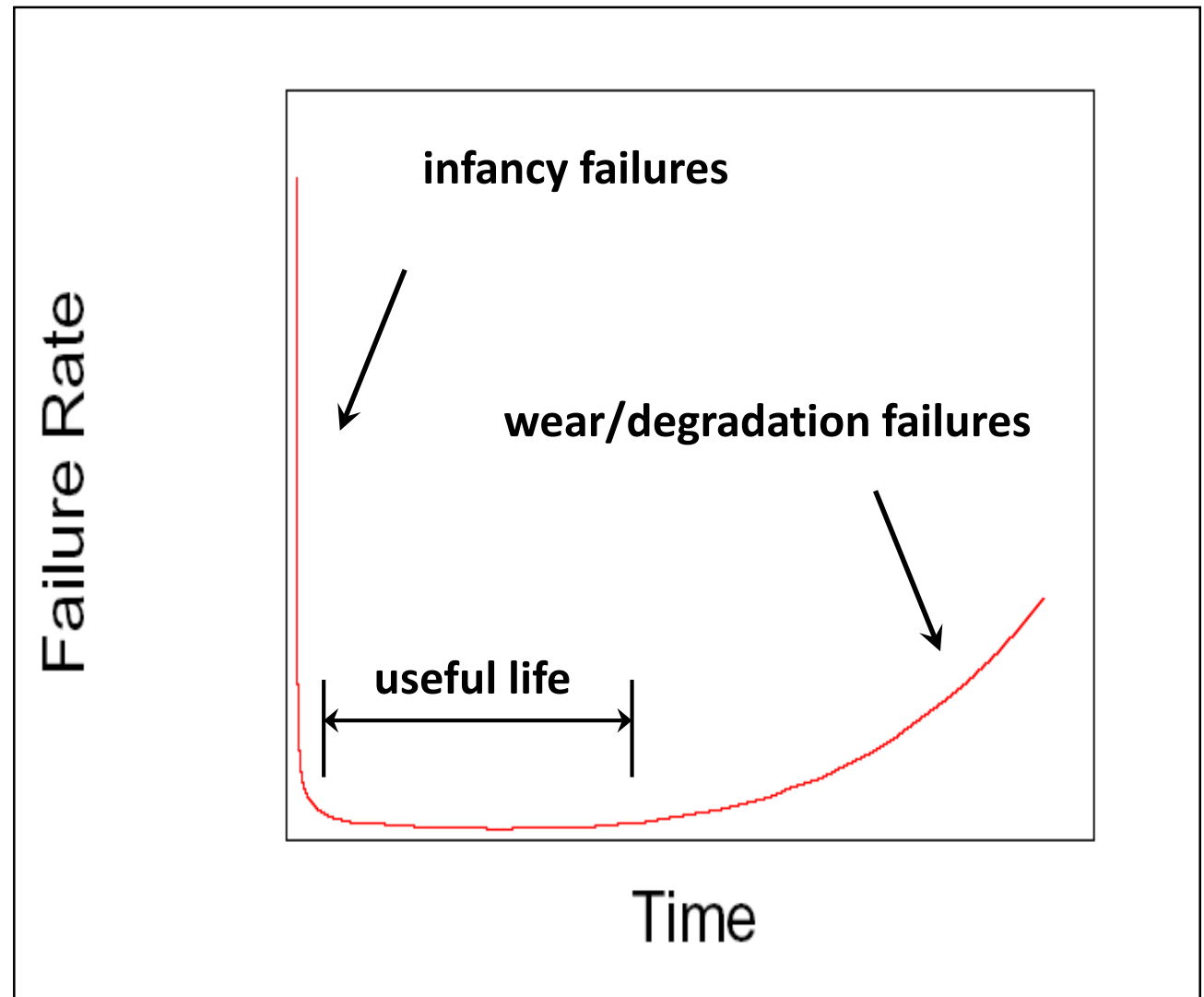


# Strength of SOFC materials

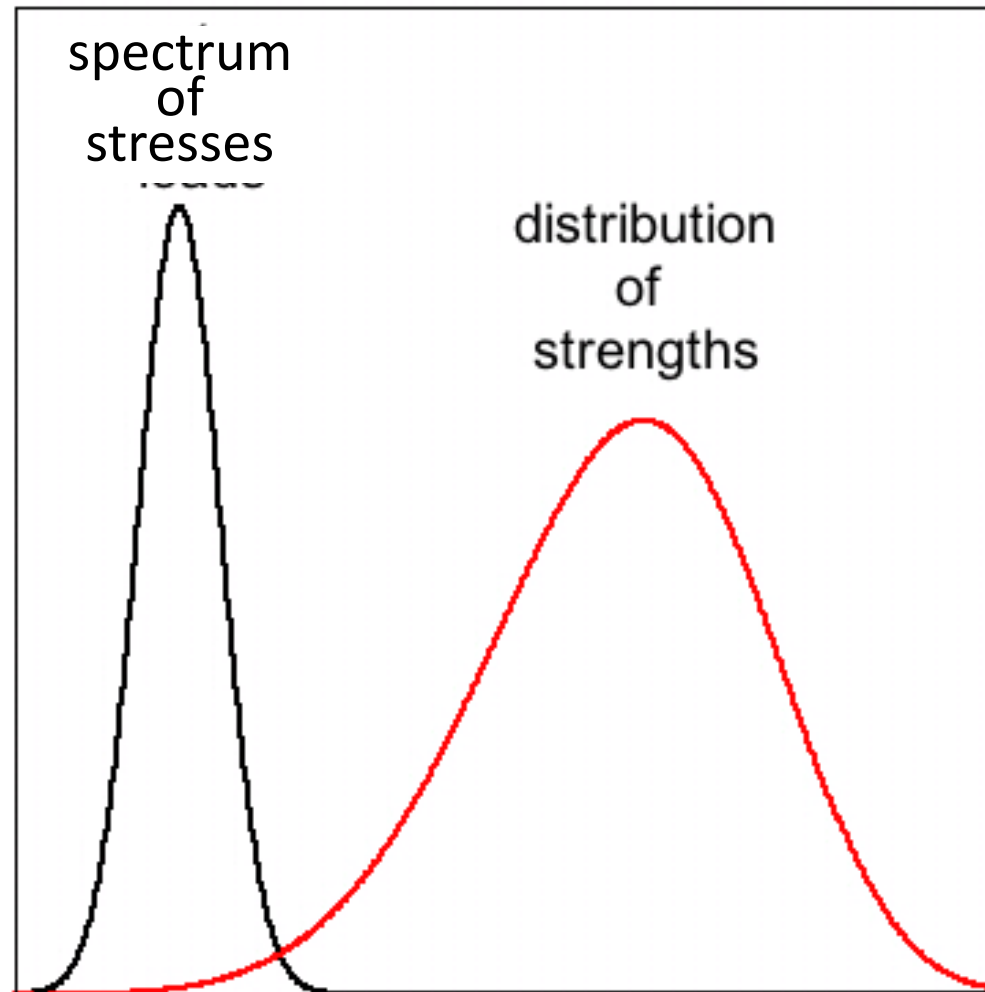


# Failure, Reliability and Durability of Systems

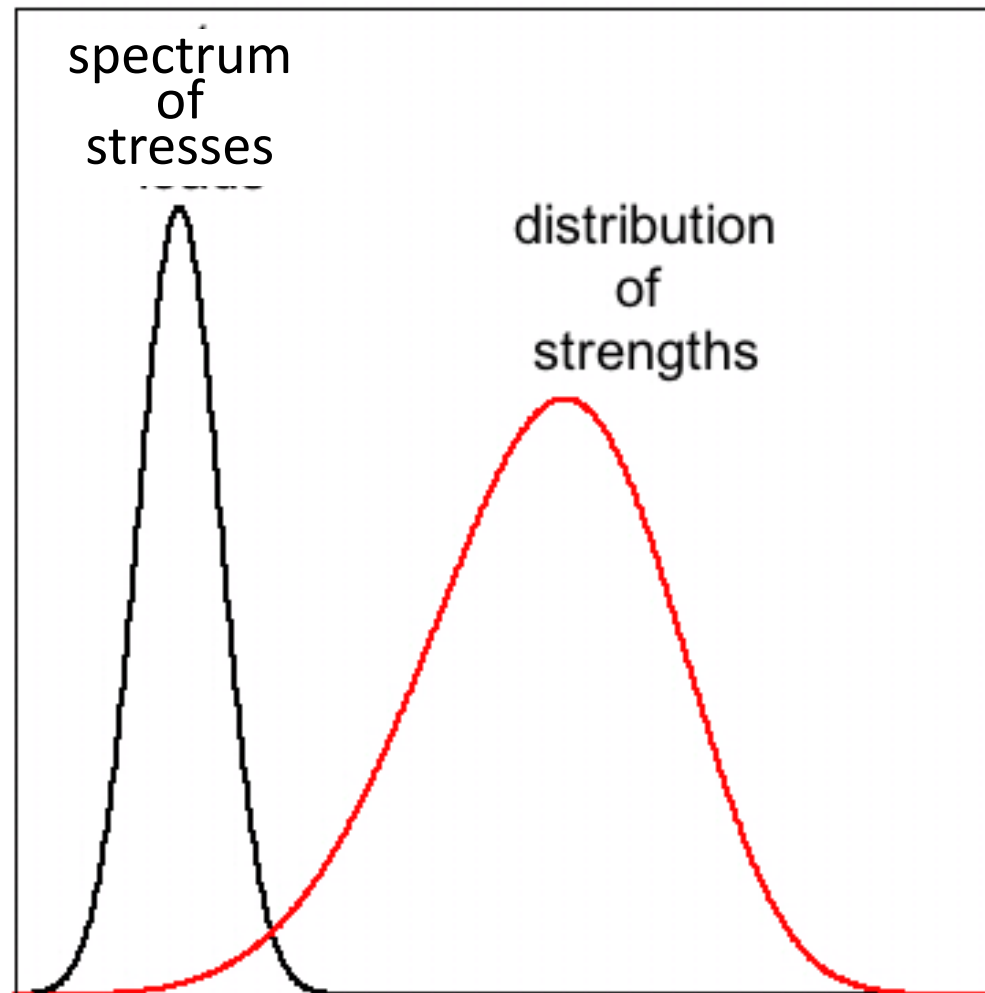
The failure rate of complex systems can be described by the bathtub curve



# Failure, Reliability and Durability of Systems

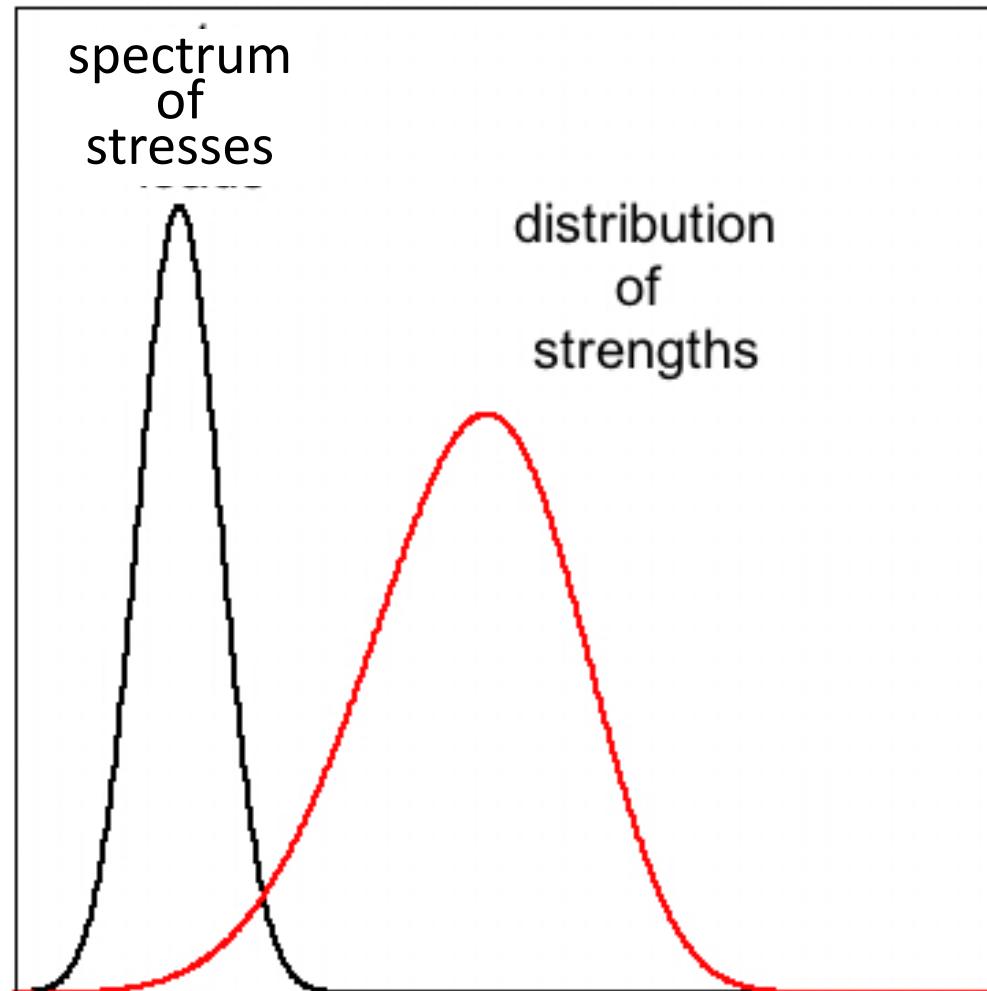


# Failure, Reliability and Durability of Systems

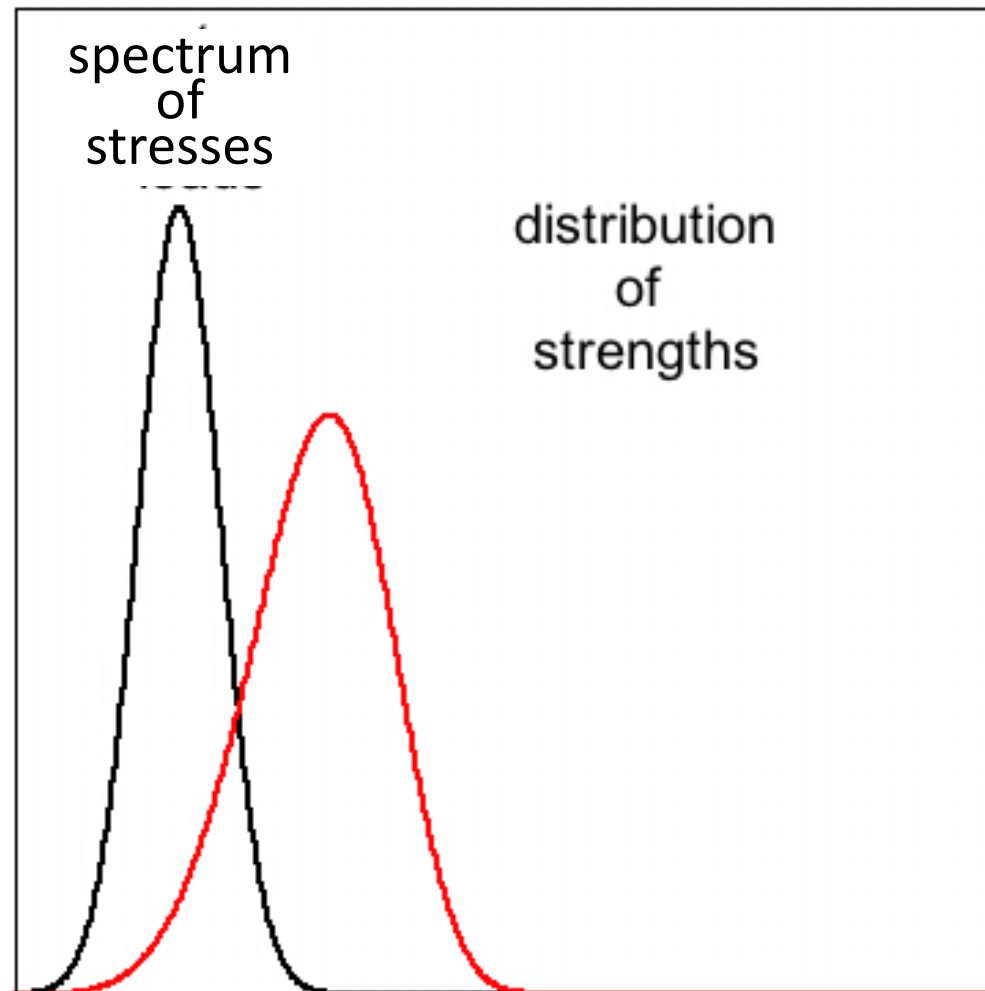




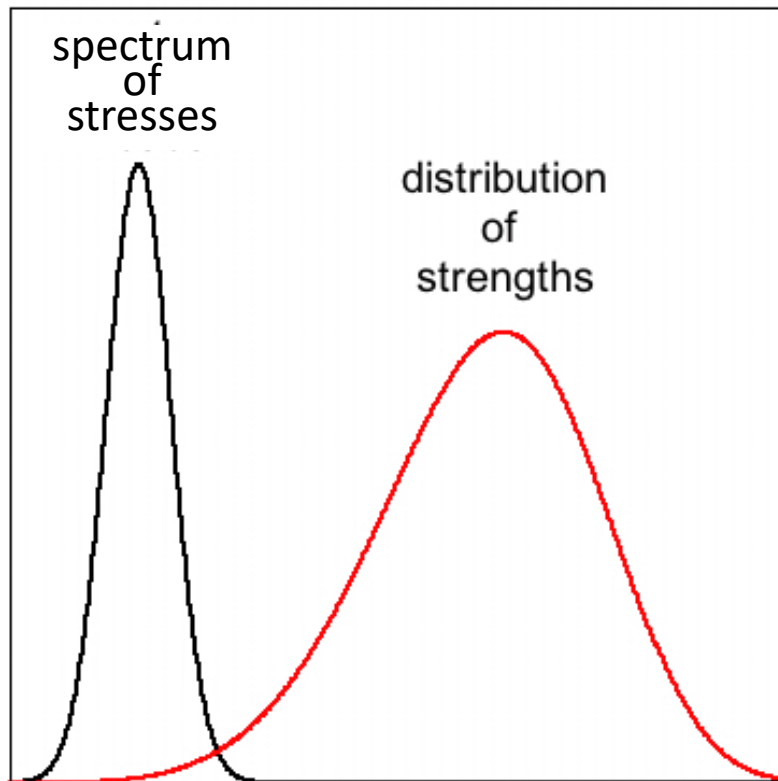
# Failure, Reliability and Durability of Systems



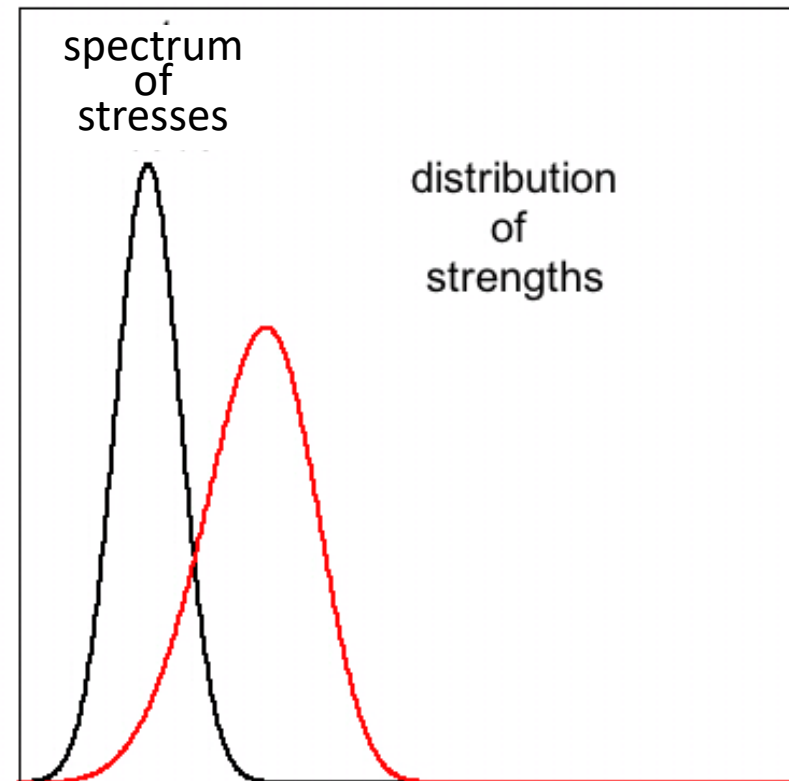
# Failure, Reliability and Durability of Systems



# Failure, Reliability and Durability of Systems



before



after

# Background

---

- The reliability and durability of materials and components for solid-oxide fuel cells is determined by their state of stress, which consists of the superposition of:
  - Residual stresses
  - Assembly stresses
  - Operational stresses
- Ni-YSZ exhibits creep deformation at temperatures relevant to the operation of SOFCs

# Questions

---

- Does creep deformation change the microstructure of anode materials?
  - If yes, how do these changes affect the functionality of the anode?
- If the layers bonded to the anode in a cell (e.g., electrolyte and interconnect) have greater creep resistance than the anode, how do stresses experienced by the anode get redistributed to the neighboring layers during SOFC operation?

# Objective

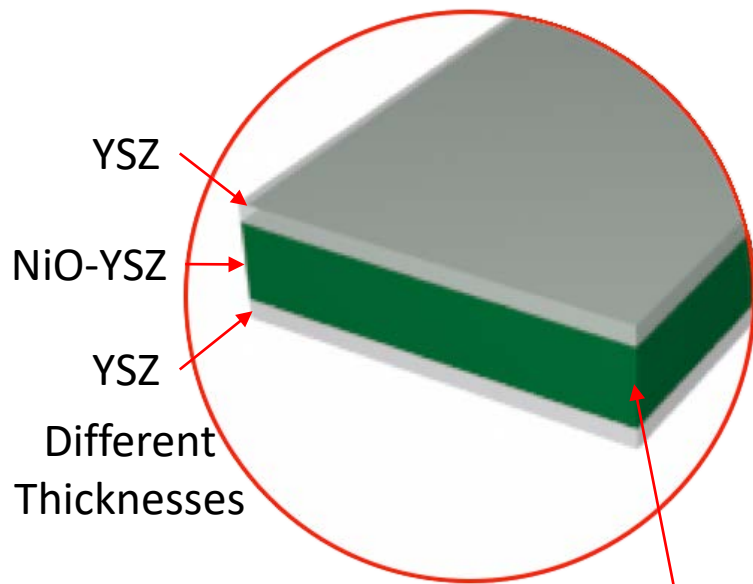
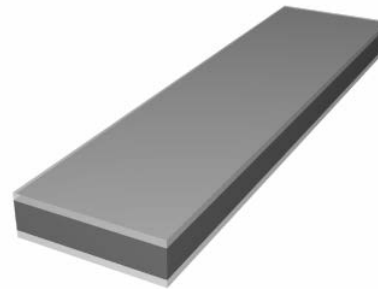
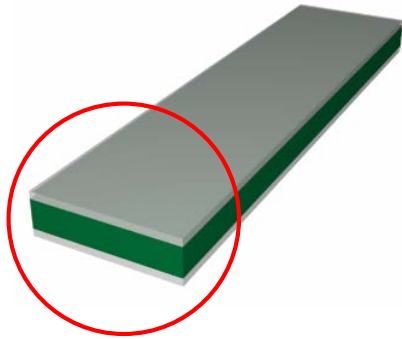
---

To understand the effect of creep deformation of anode materials on its microstructure and on the redistribution of stresses in SOFCs, and to determine the implications on their durability and reliability

# Materials (Sandwich Configuration)

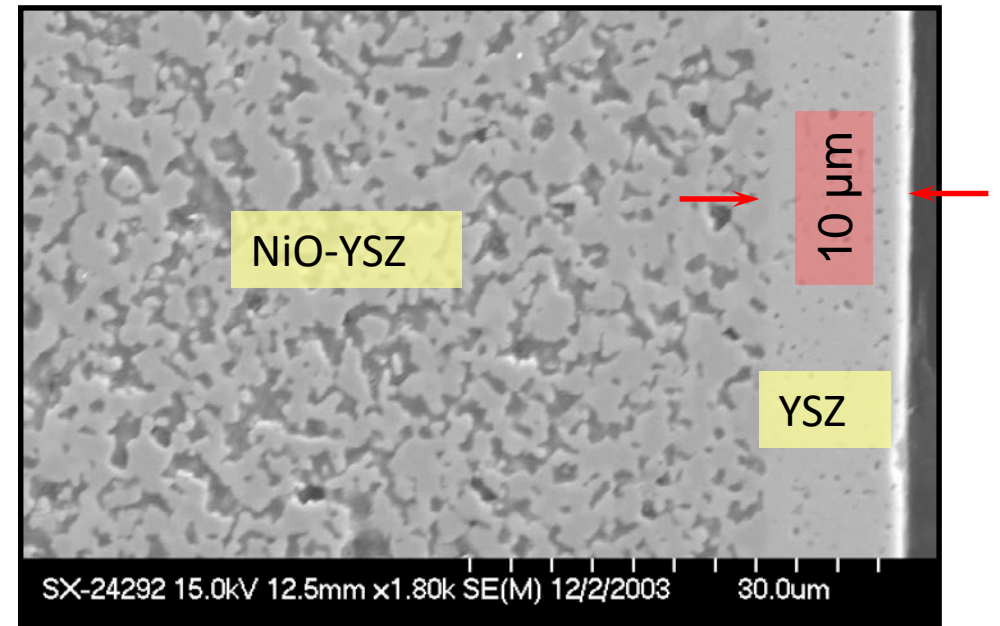
As-processed

After reduction



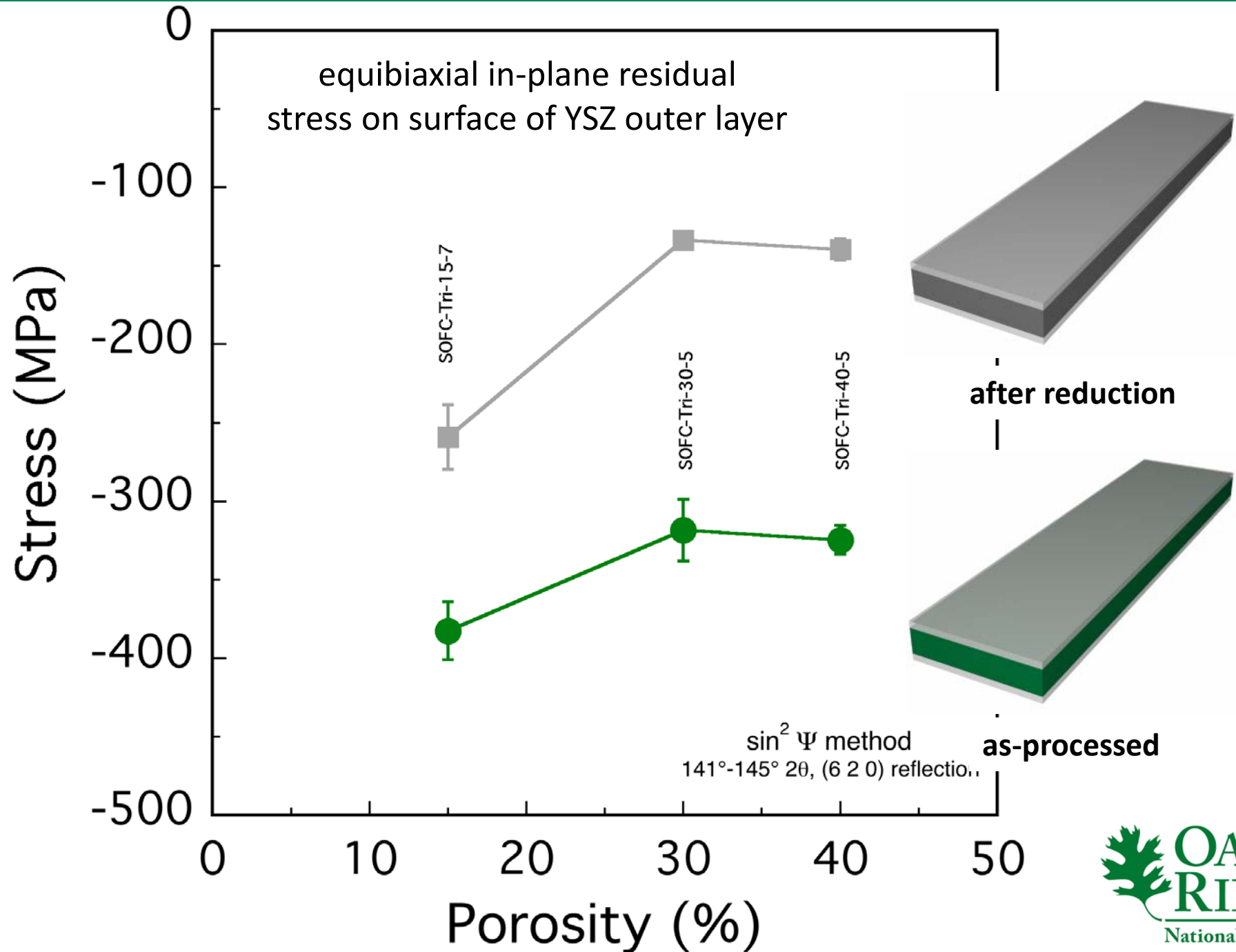
Porosity

- 15%
- 30%
- 40%



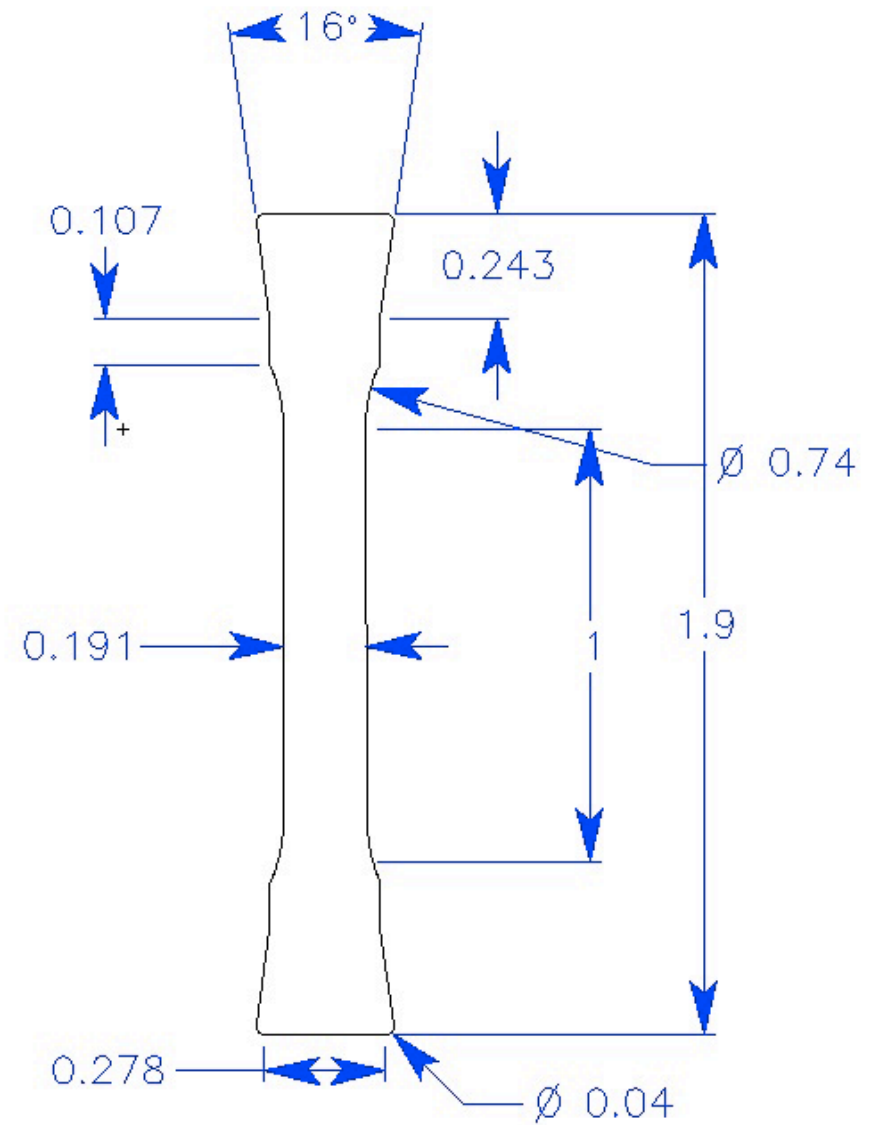
# Residual Stresses

Measurements obtained on surface of YSZ layer by X-ray diffraction

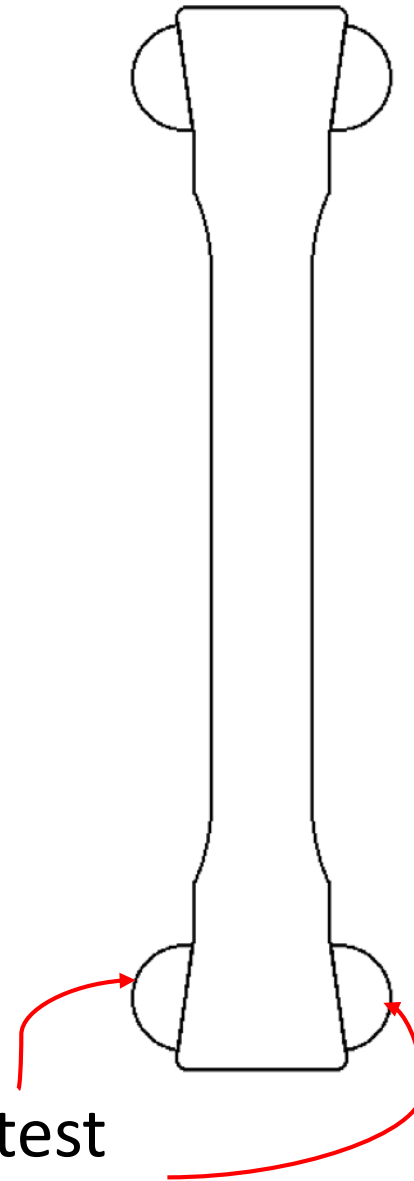




# Shoulder-loaded Tensile Specimen



# Creep Testing Facility



Load is transferred to the test specimen through its shoulders

Lara-Curzio et al. June 13, 2017

# Creep Testing Facility

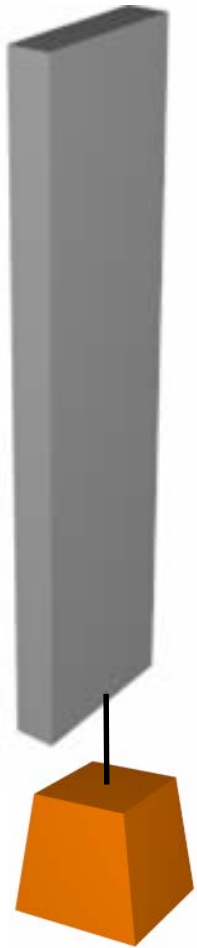


- High temperature
- Controlled environment

Lara-Curzio et al. June 13, 2017

# Creep Deformation

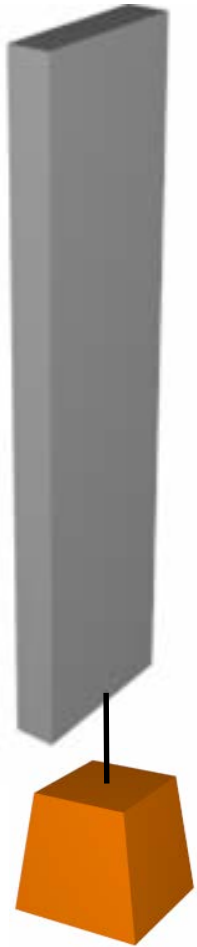
---



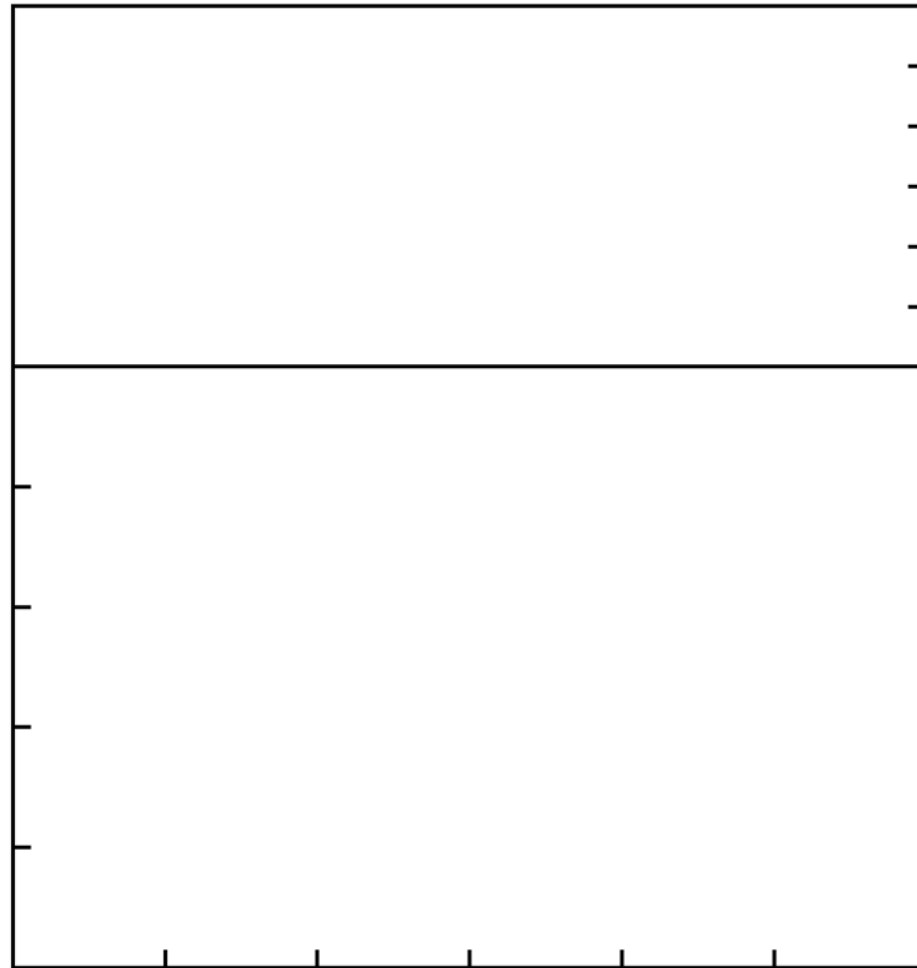
**Test Specimen**

**Constant  
Stress**

# Creep Deformation



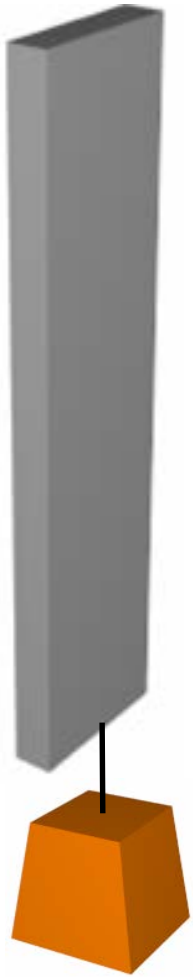
strain



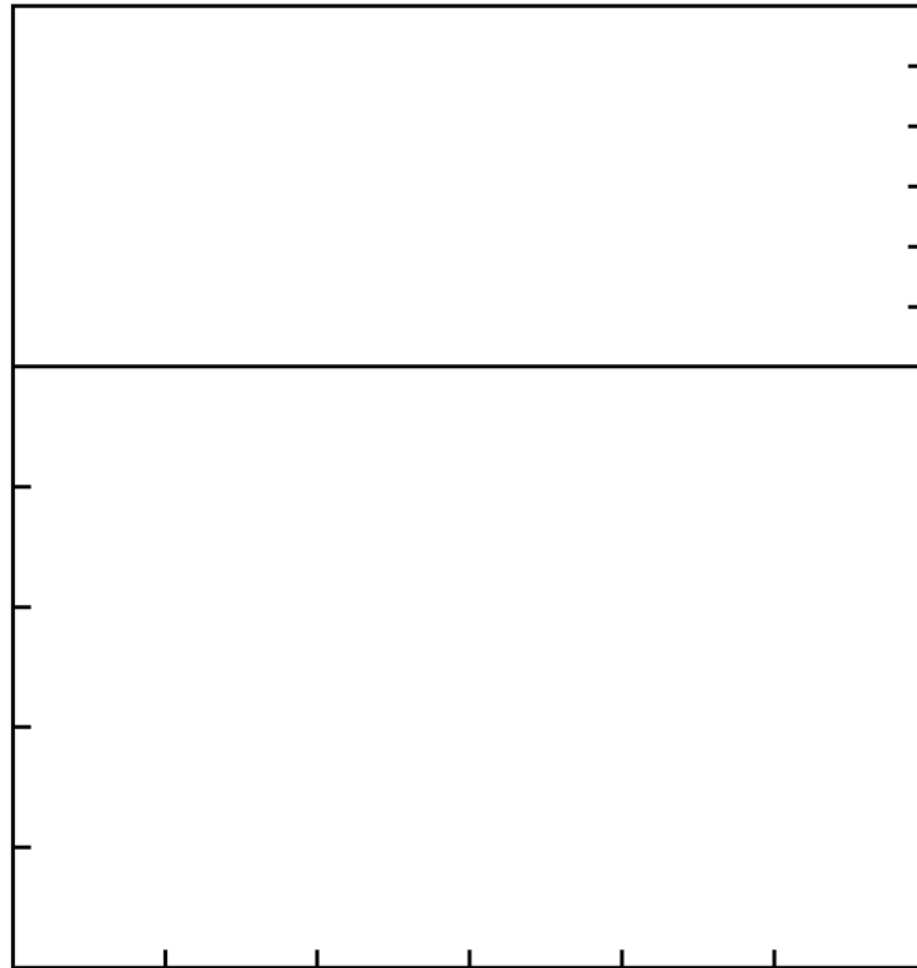
strain rate (s<sup>-1</sup>)

Time

# Creep Deformation



strain



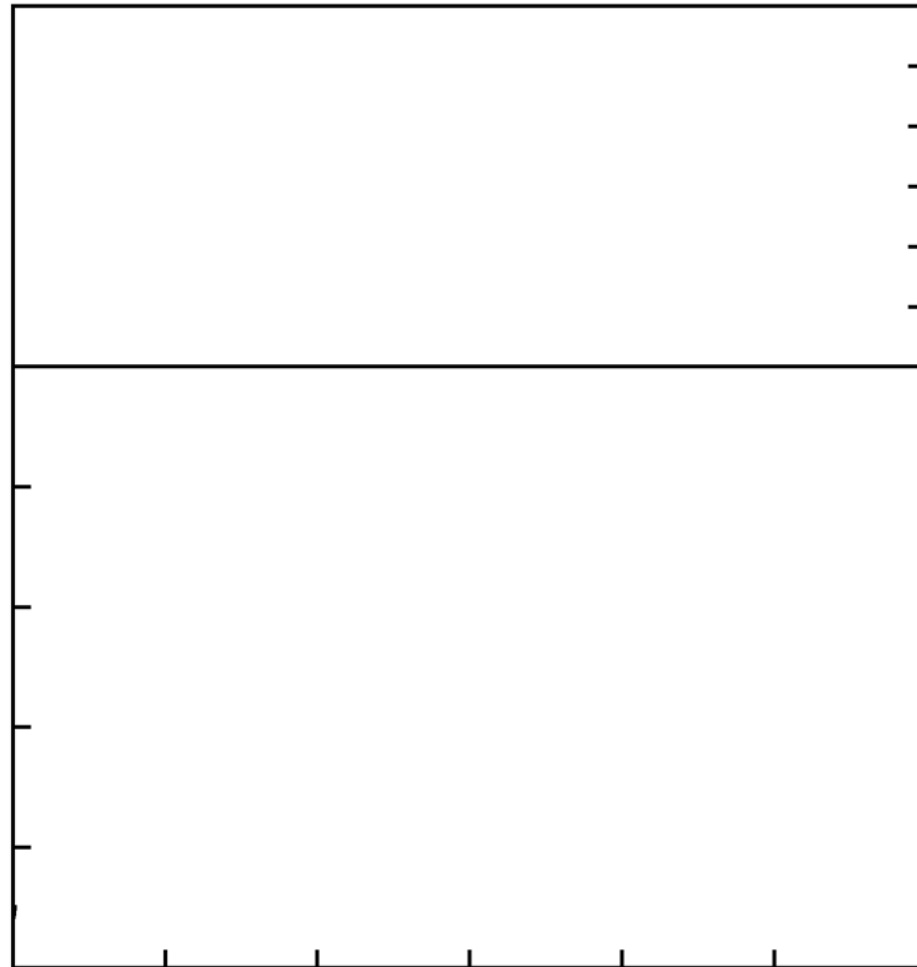
Time

strain rate ( $s^{-1}$ )

# Creep Deformation



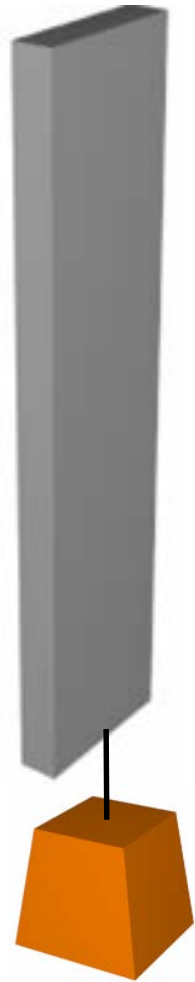
strain



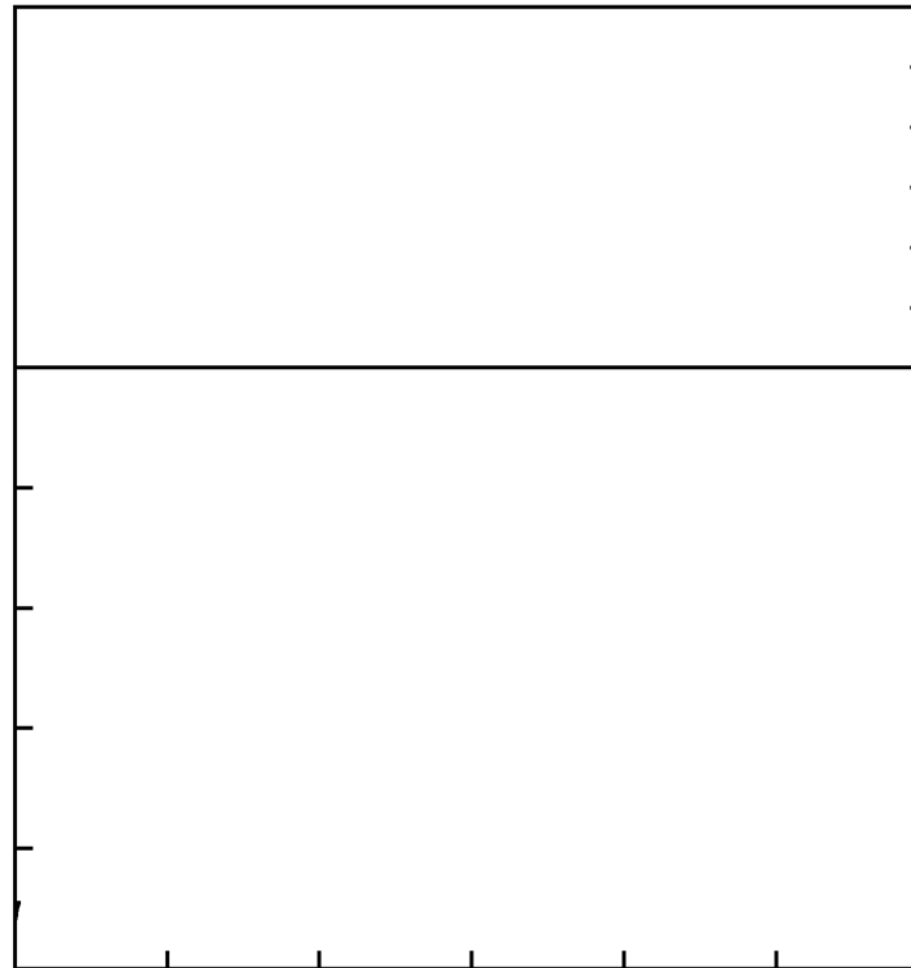
strain rate ( $s^{-1}$ )

Time

# Creep Deformation



strain

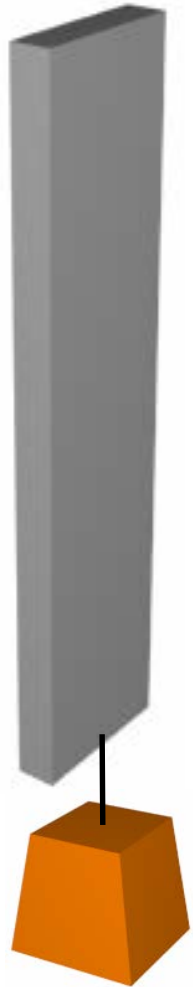


strain rate ( $s^{-1}$ )

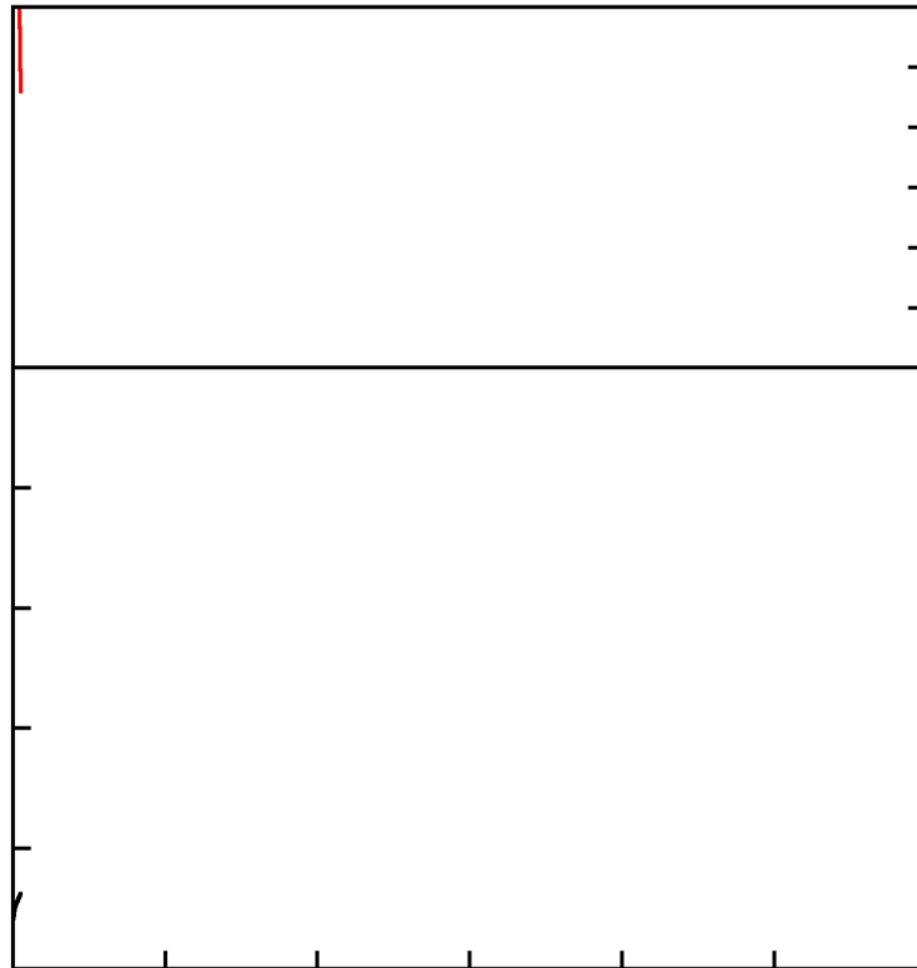
Time



# Creep Deformation



strain



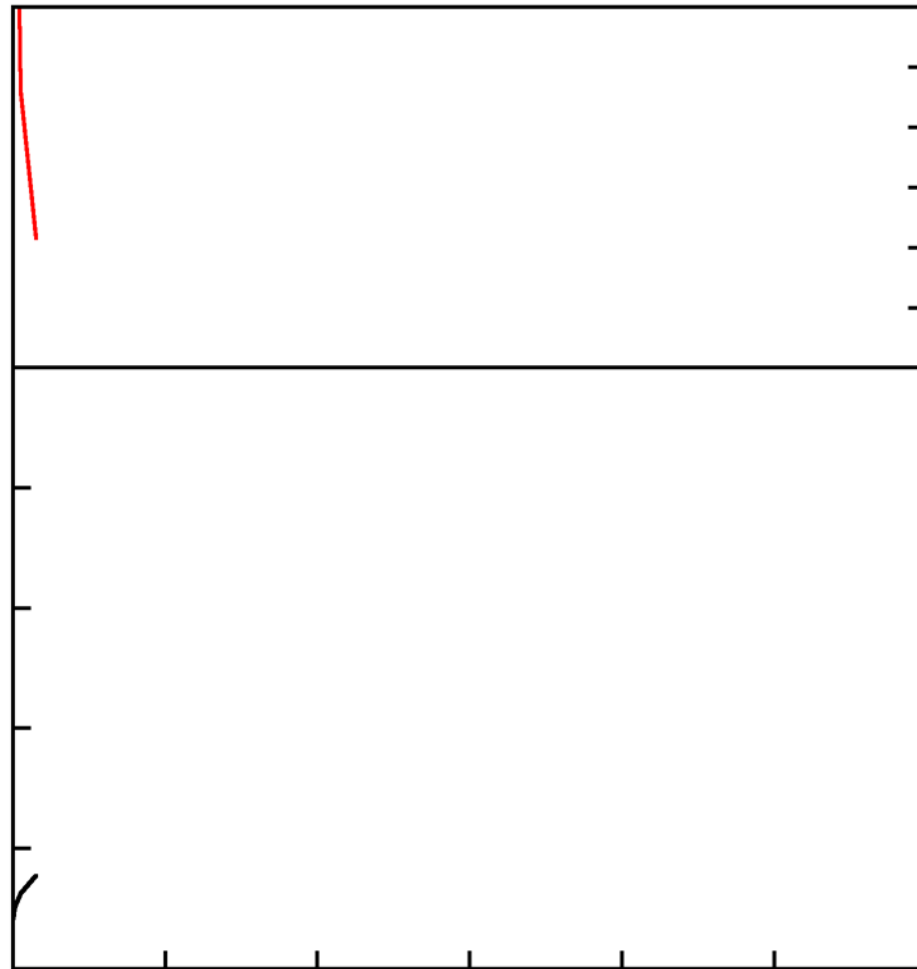
Time

strain rate ( $s^{-1}$ )

# Creep Deformation



strain

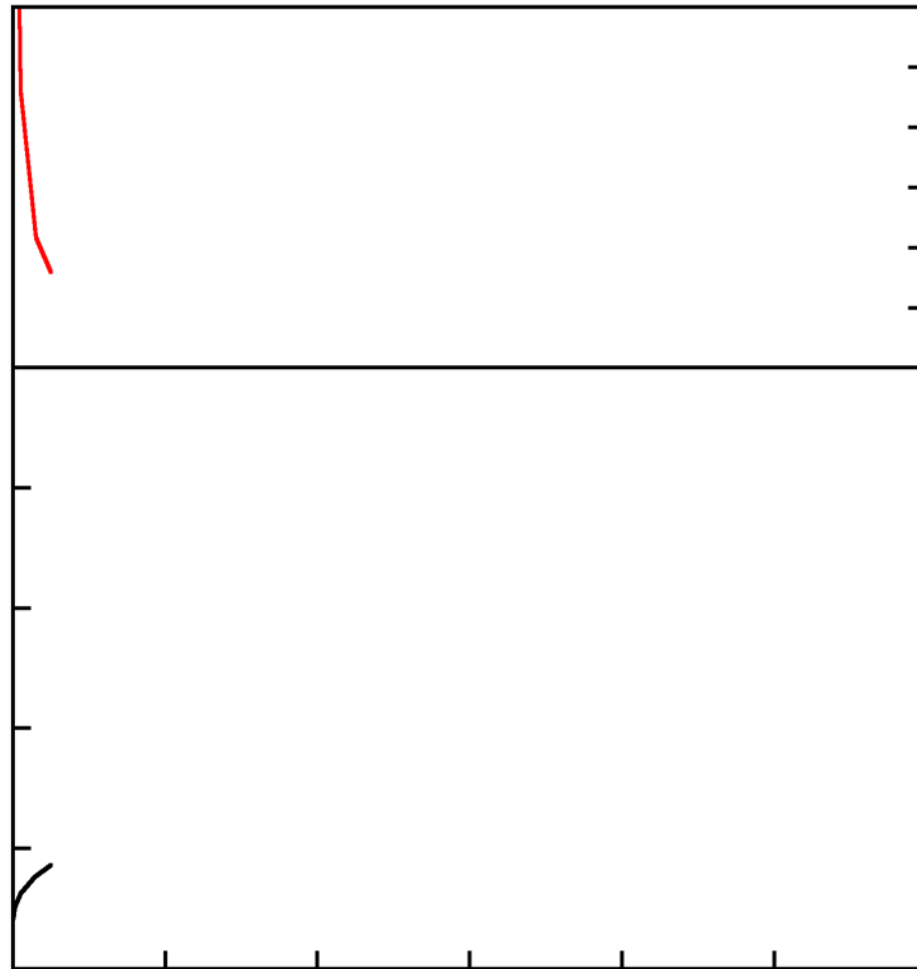


Time

# Creep Deformation

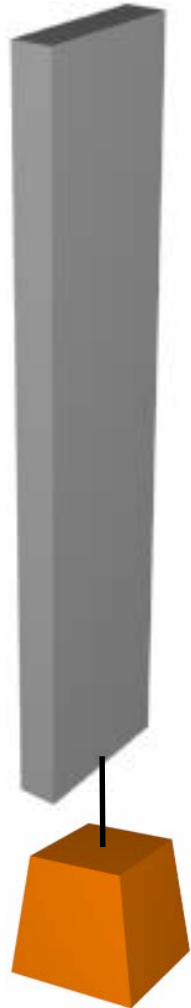


strain

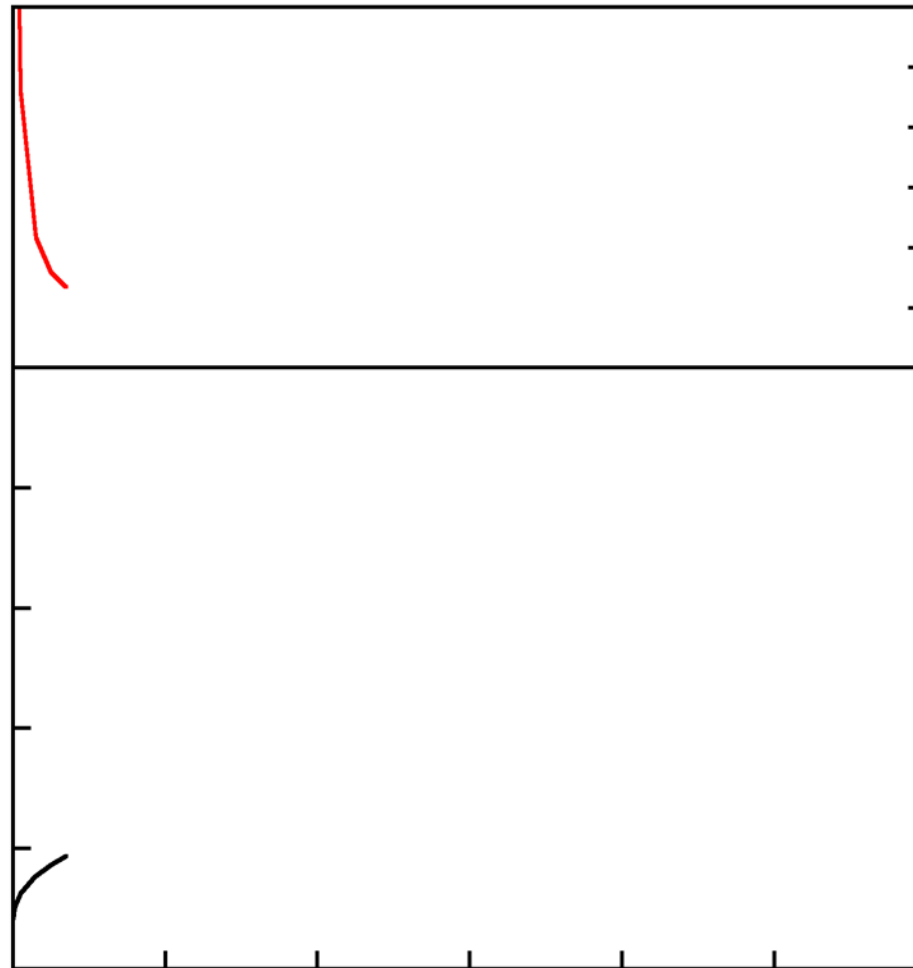


Time

# Creep Deformation



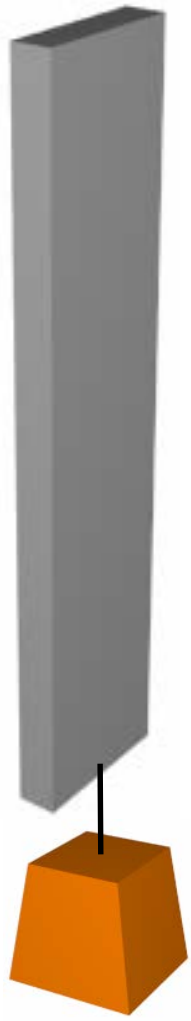
strain



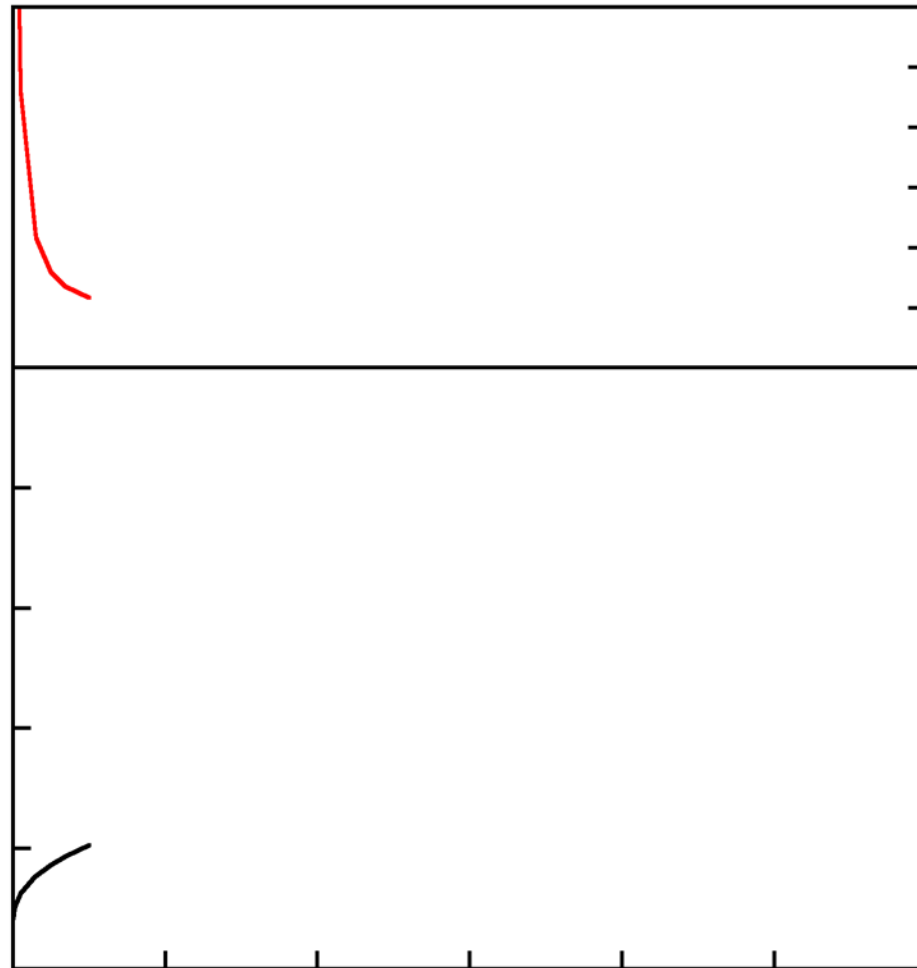
Time

strain rate ( $s^{-1}$ )

# Creep Deformation

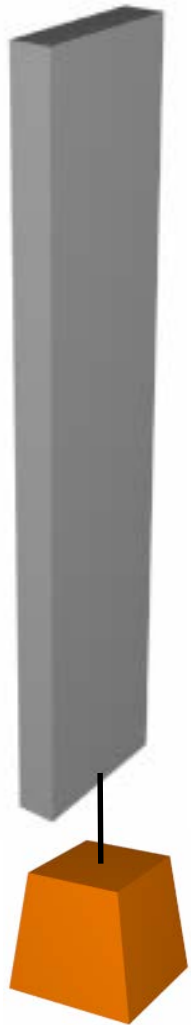


strain

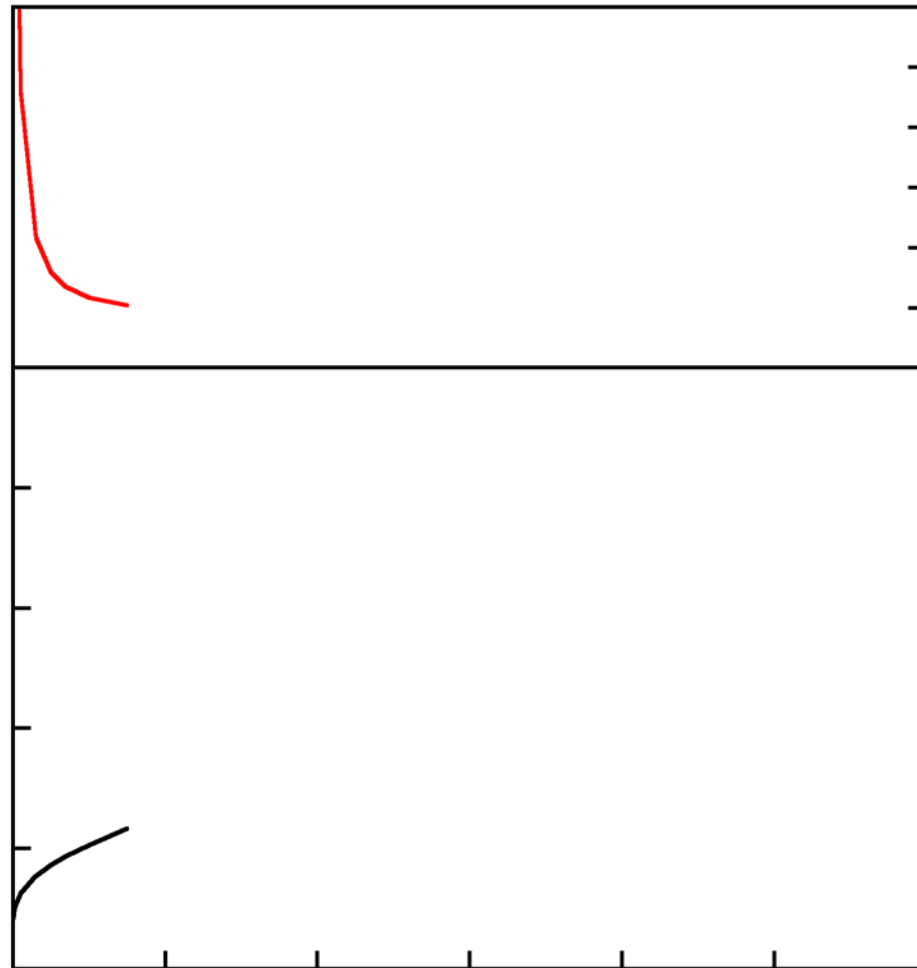


Time

# Creep Deformation



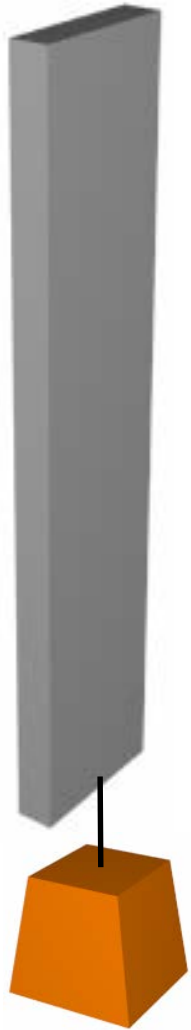
strain



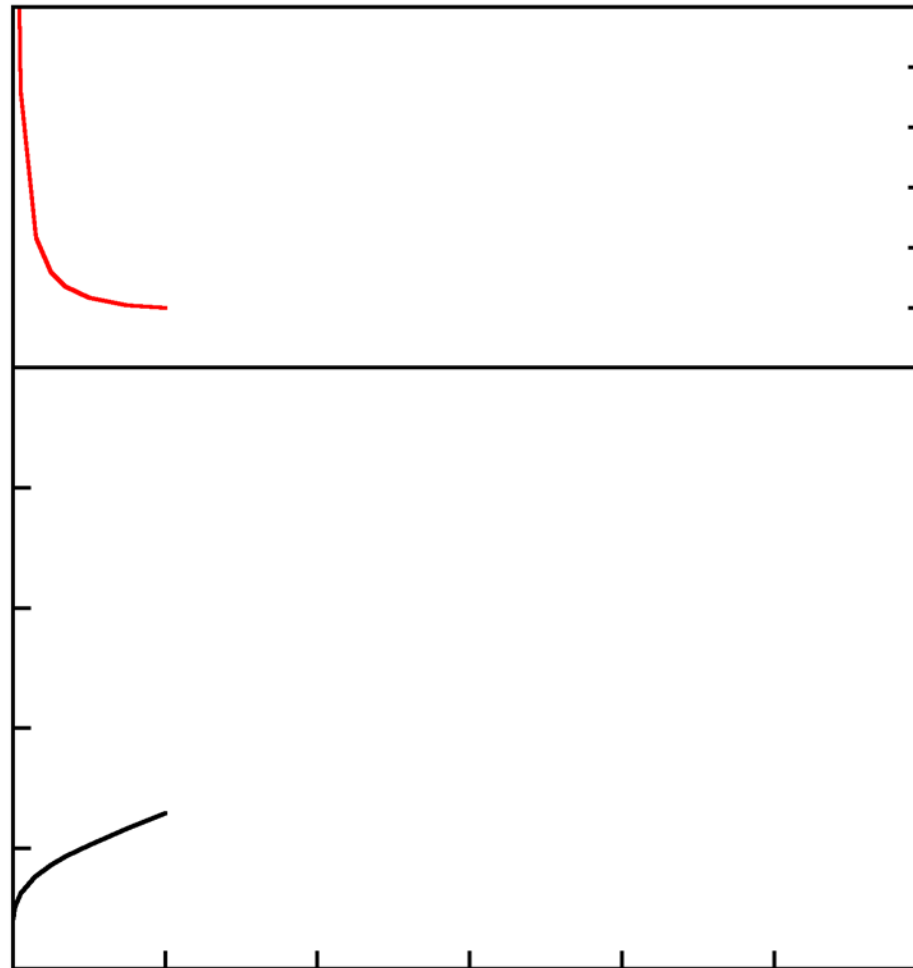
strain rate (s<sup>-1</sup>)

Time

# Creep Deformation



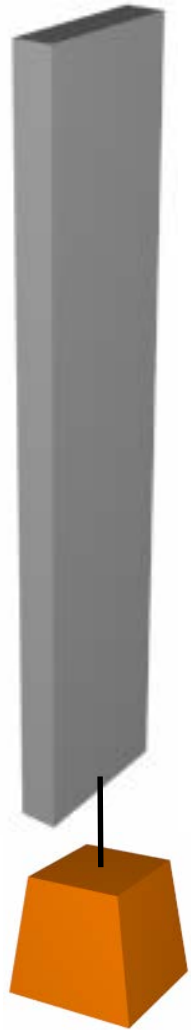
strain



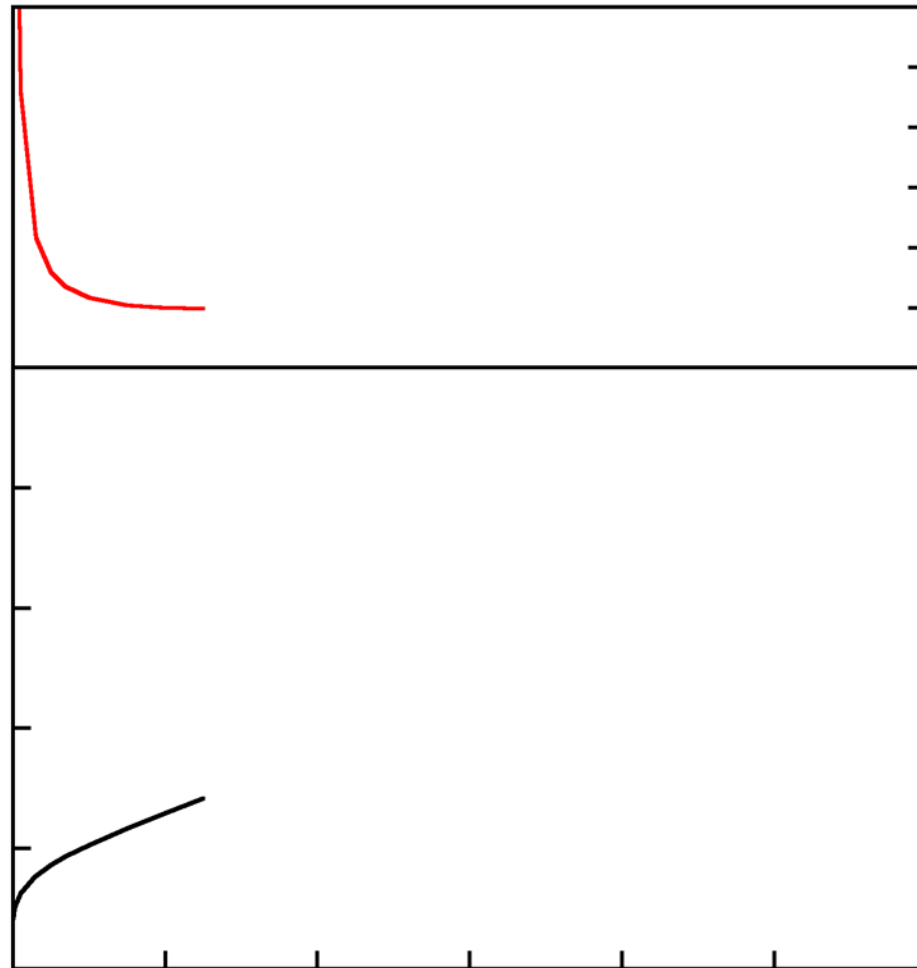
strain rate (s<sup>-1</sup>)

Time

# Creep Deformation



strain

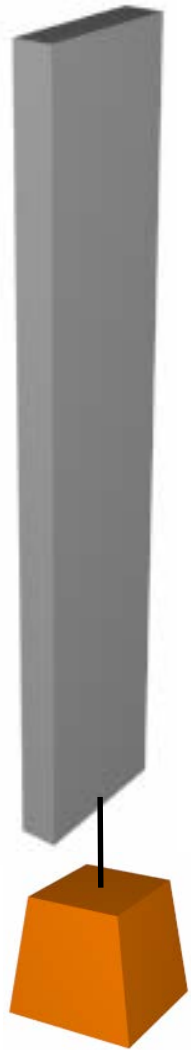


Time

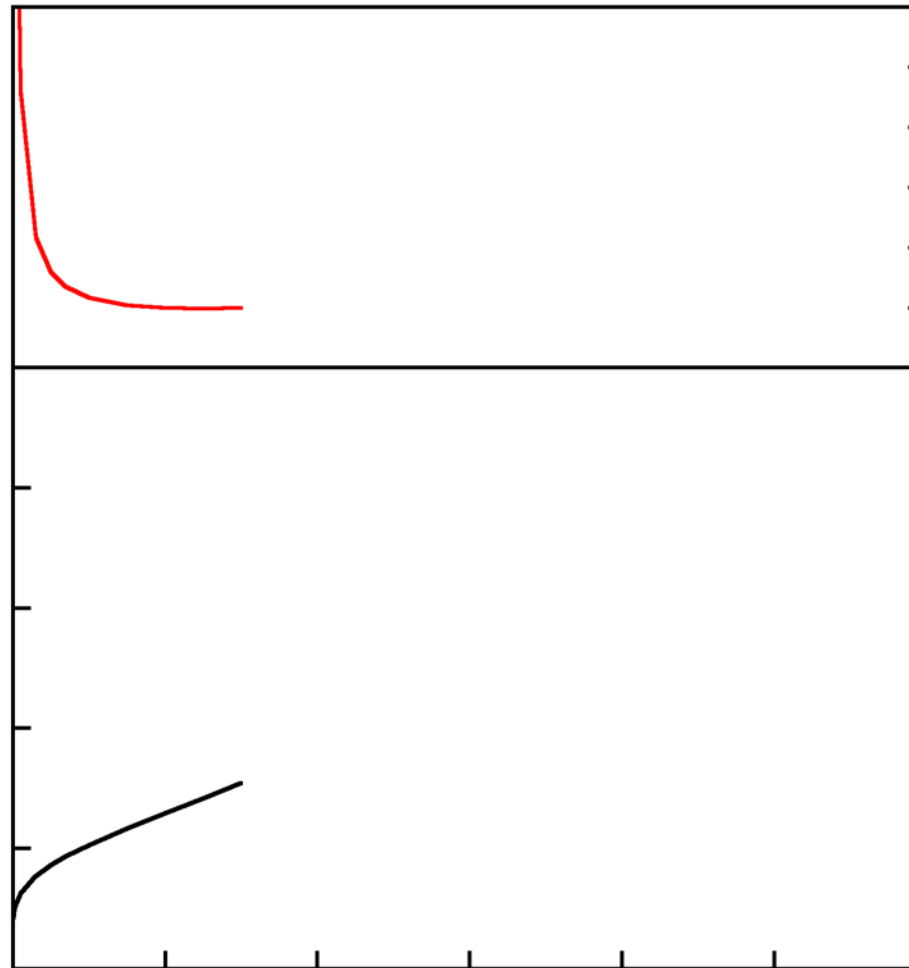
strain rate ( $s^{-1}$ )



# Creep Deformation



strain



Time

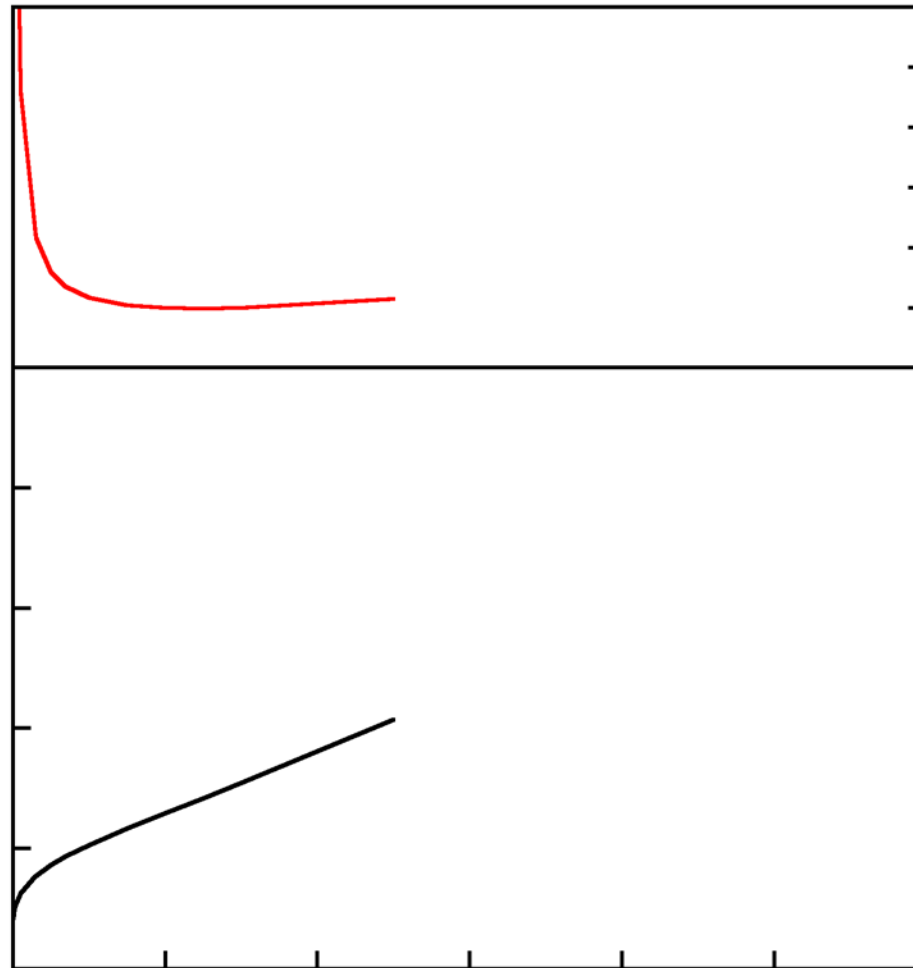
strain rate ( $s^{-1}$ )



# Creep Deformation



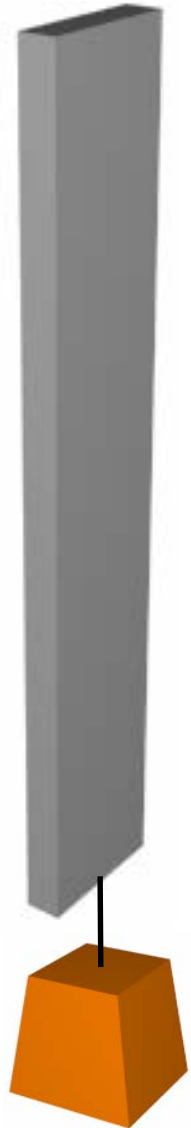
strain



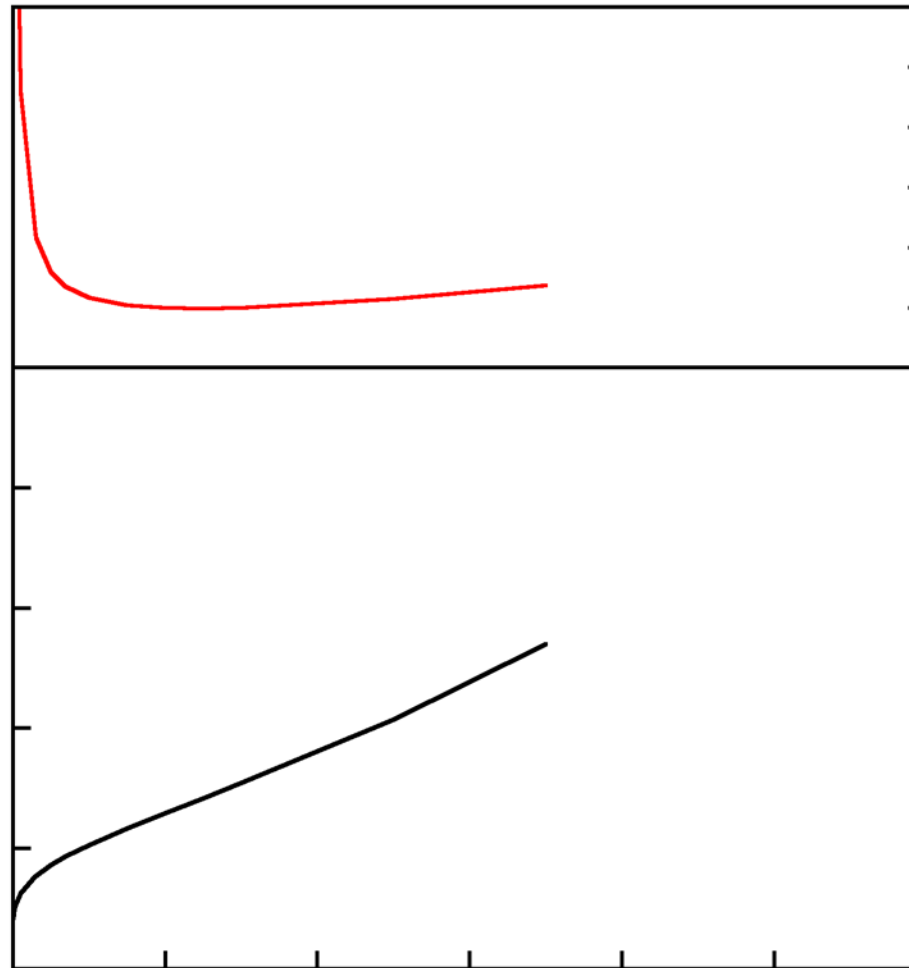
Time

strain rate ( $s^{-1}$ )

# Creep Deformation



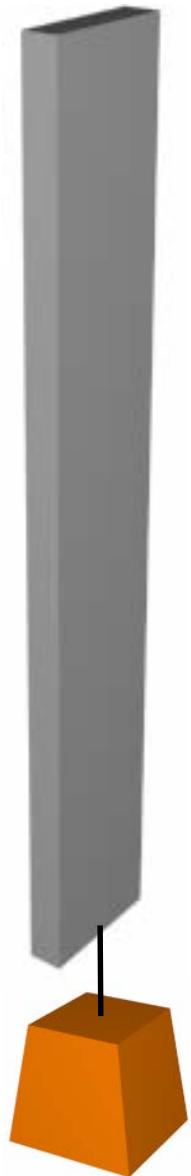
strain



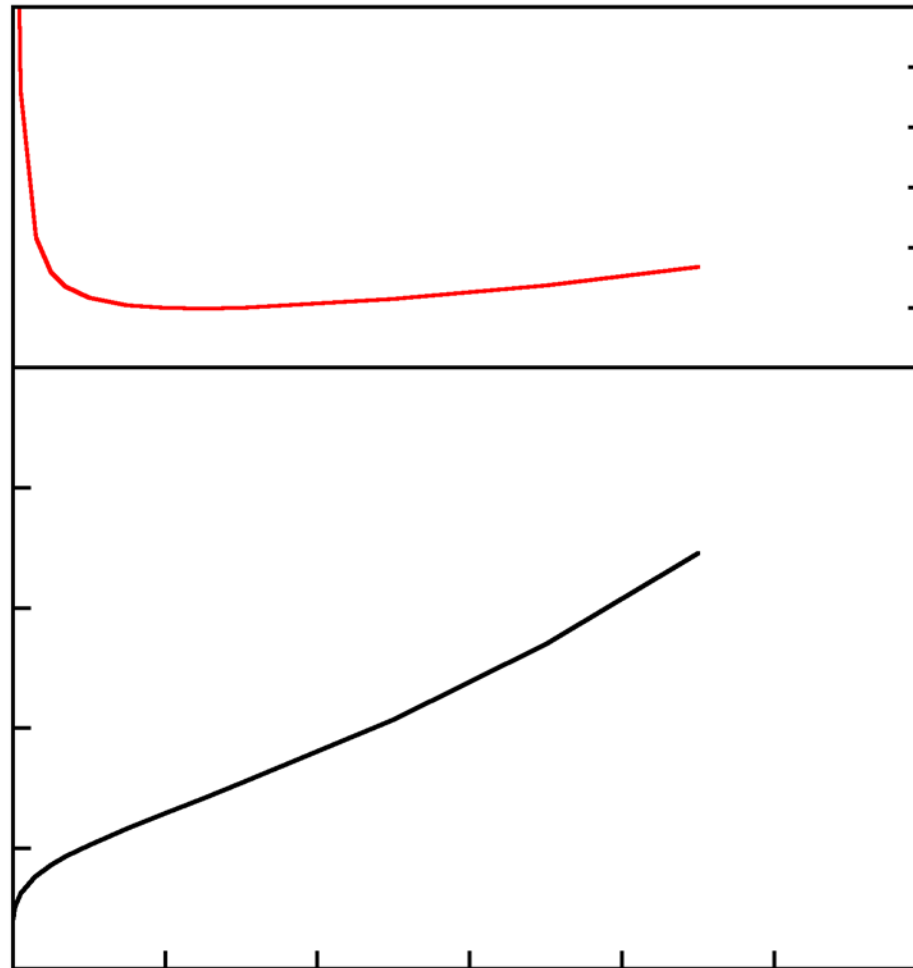
Time

strain rate ( $s^{-1}$ )

# Creep Deformation



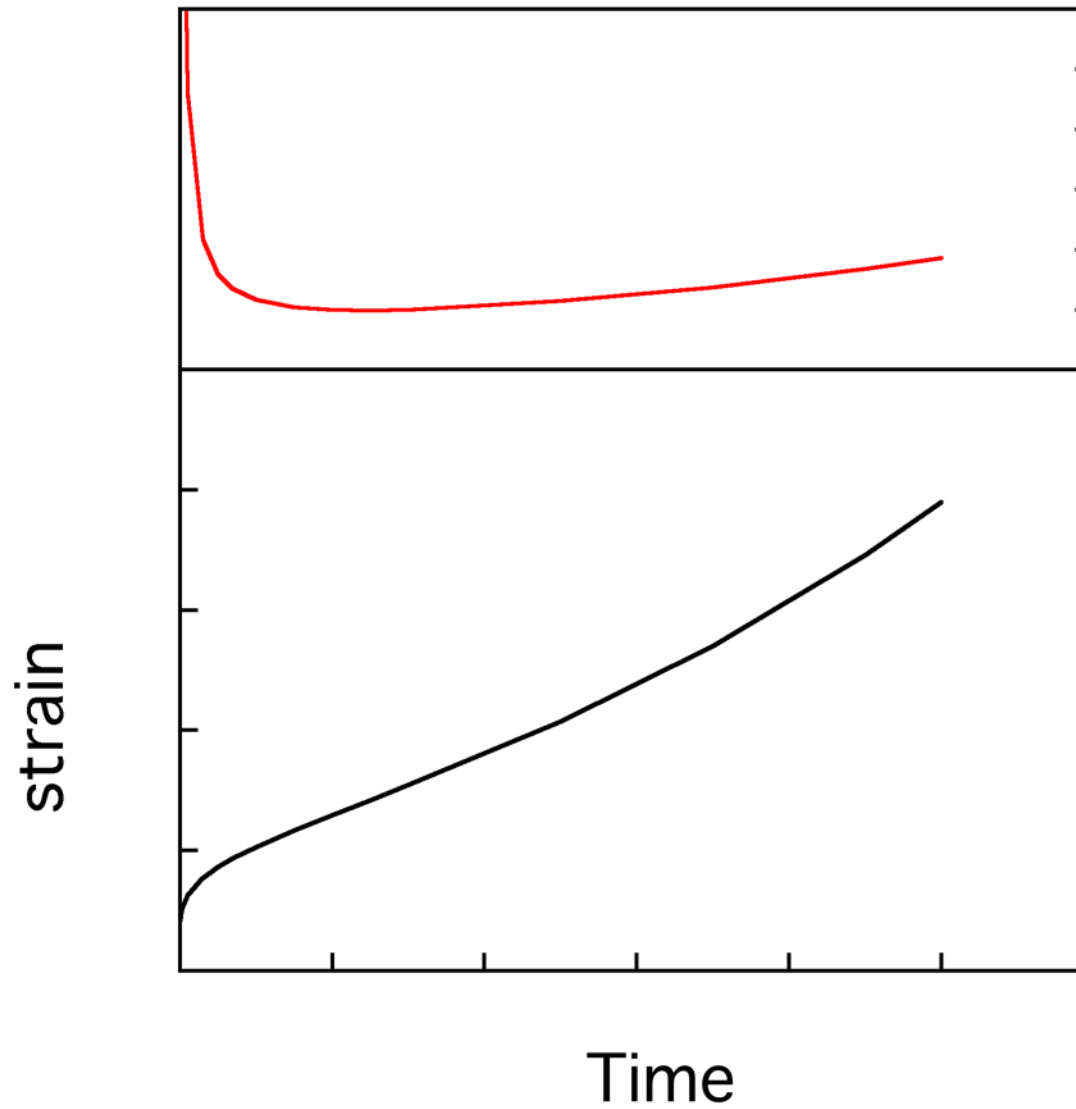
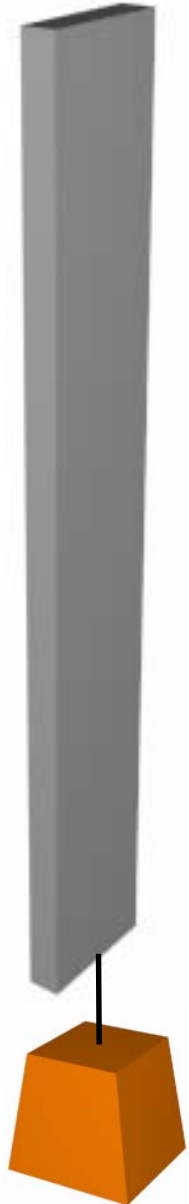
strain



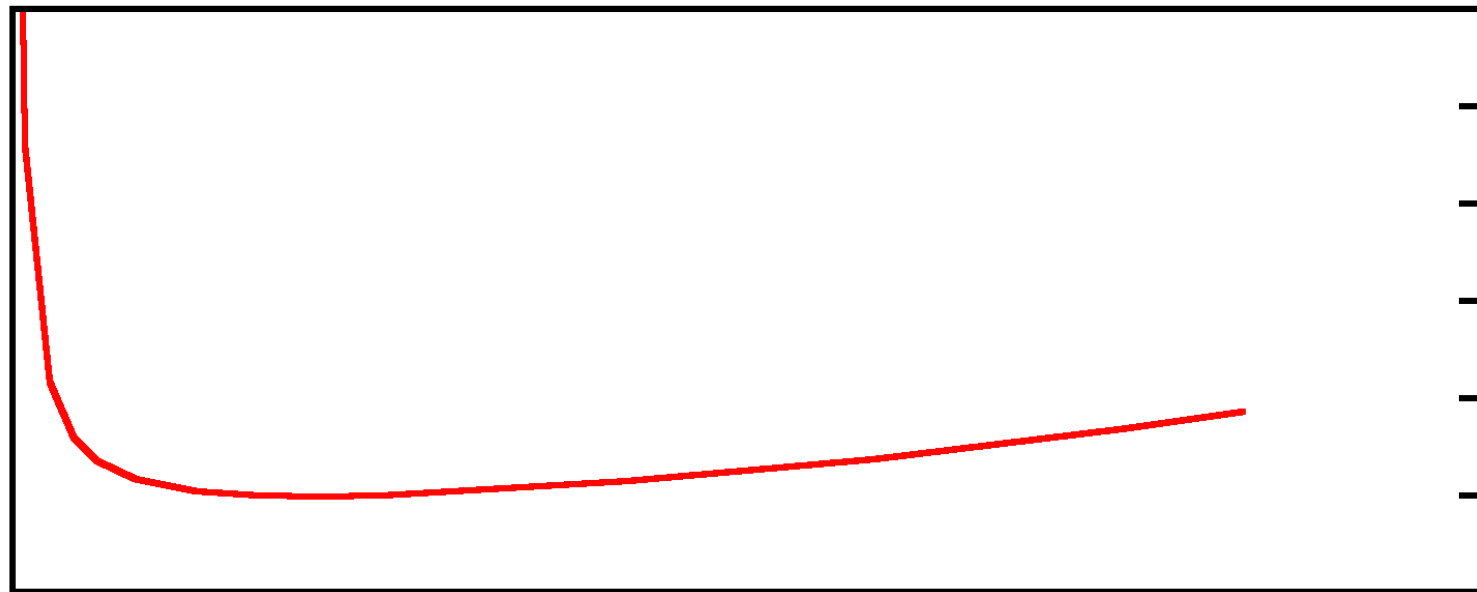
Time

strain rate ( $s^{-1}$ )

# Creep Deformation



# Creep Deformation



strain rate ( $s^{-1}$ )

$$\frac{d\varepsilon}{dt} = A \sigma^n e^{-\frac{Q}{RT}}$$

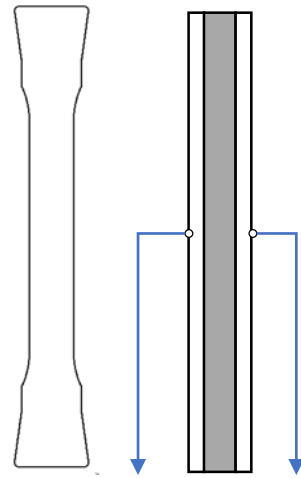
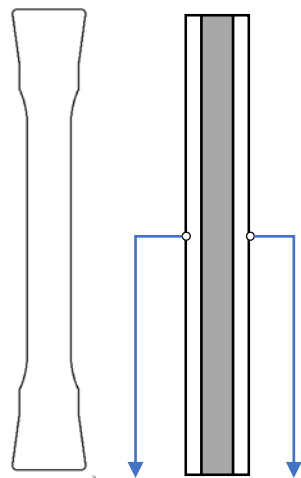
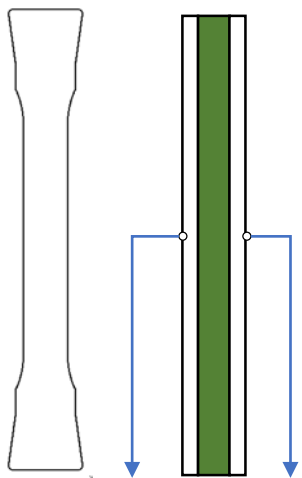
---

# Methodology (Residual Stress Measurements by XRD)

As-processed

After Reduction

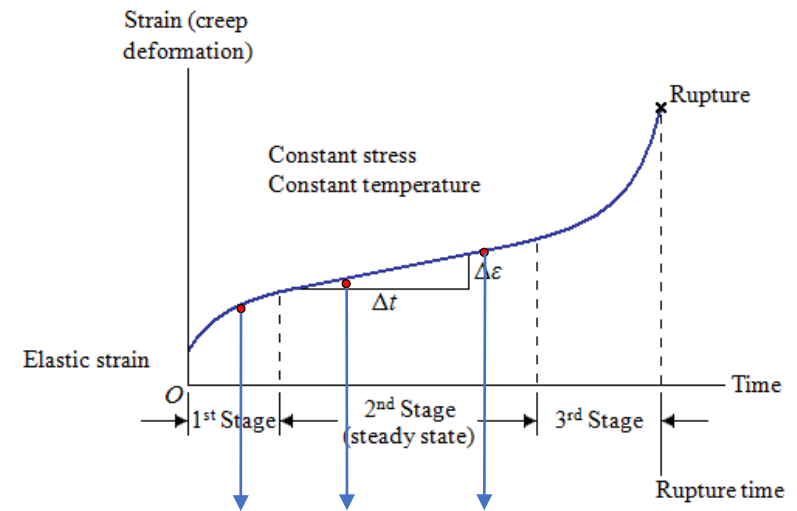
After Creep Deformation  
(by interrupting creep tests)



Residual Stress  
Measurement

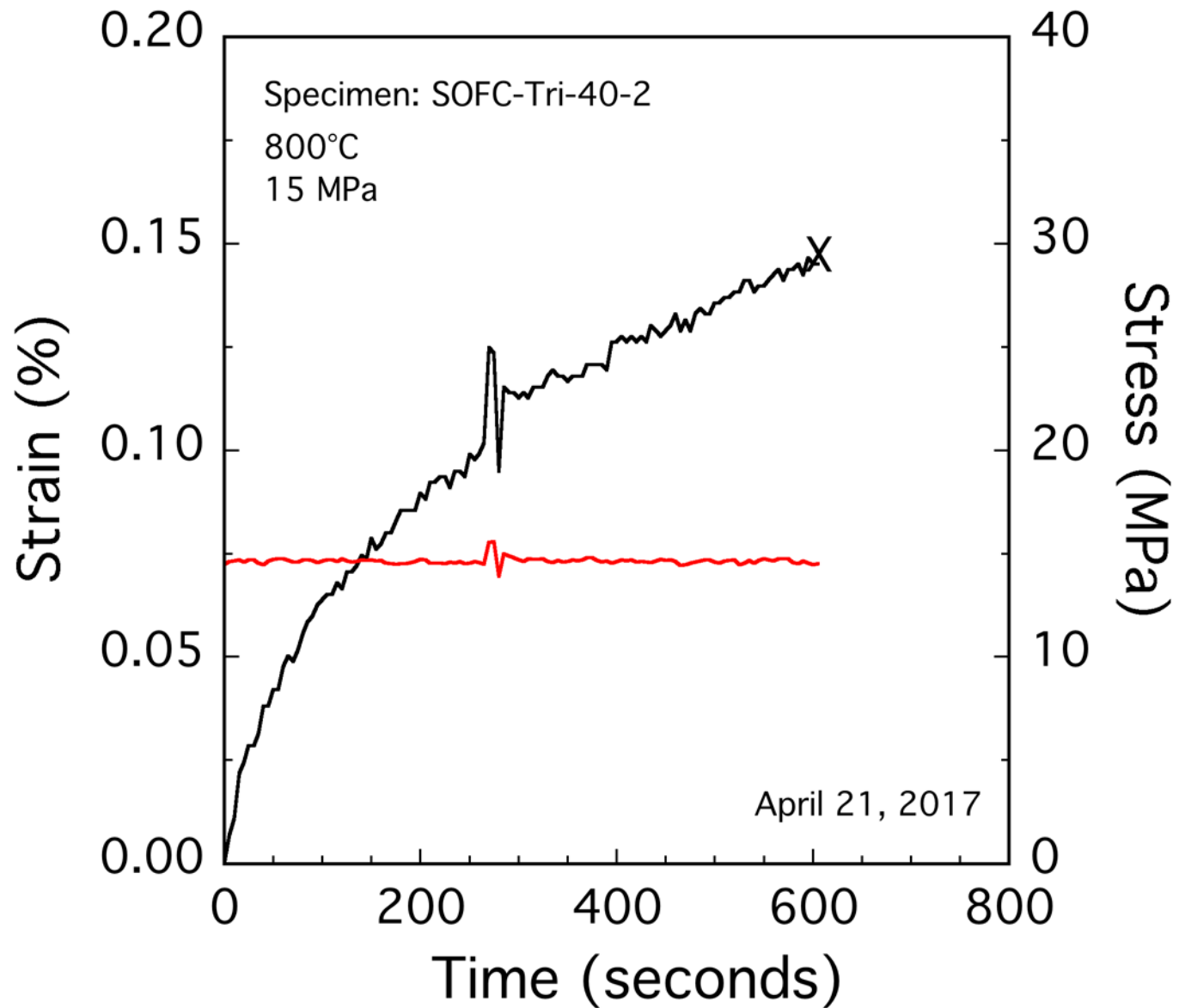
Residual Stress  
Measurement

Residual Stress  
Measurement



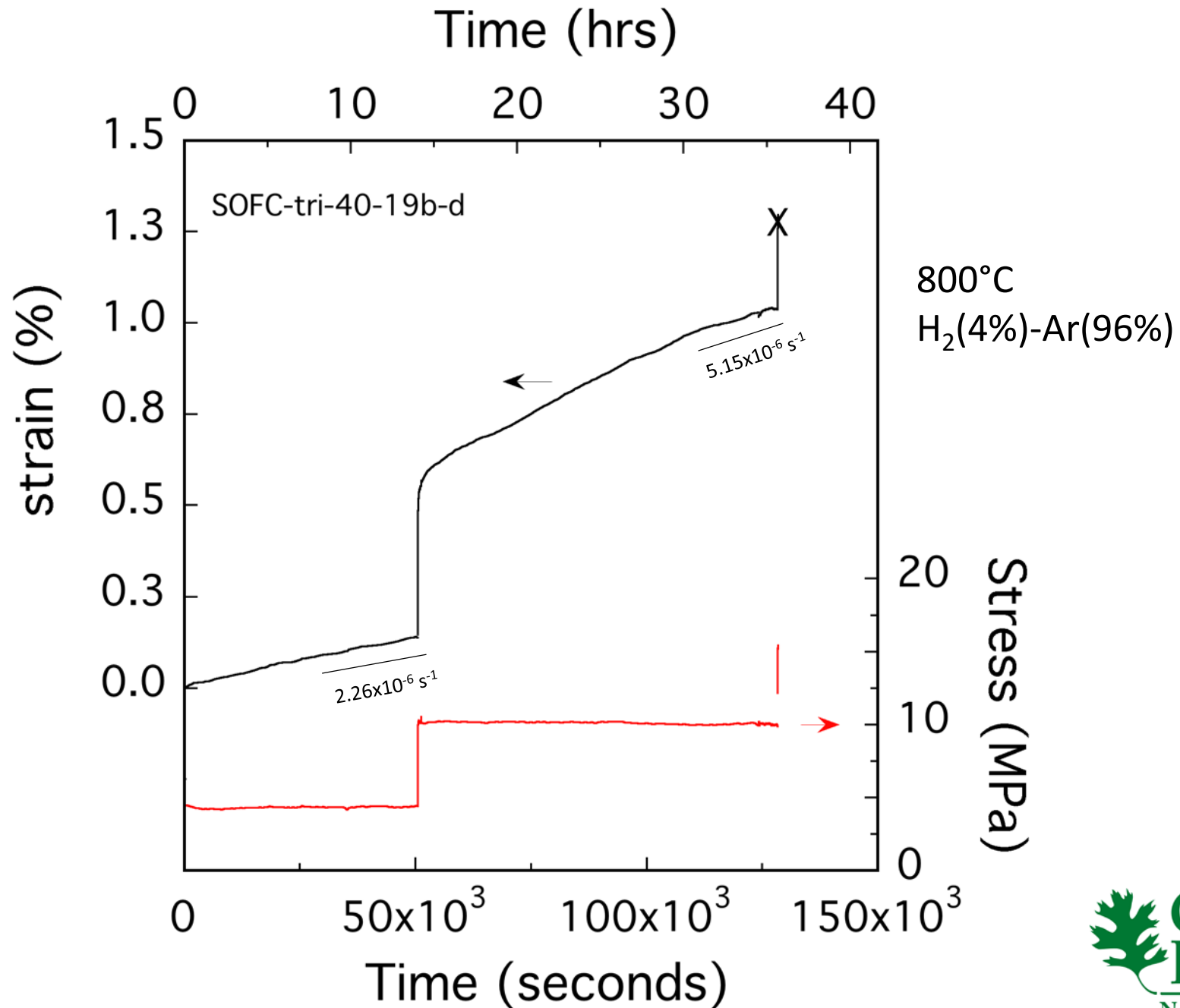
Interrupted creep test

# Creep Testing Results



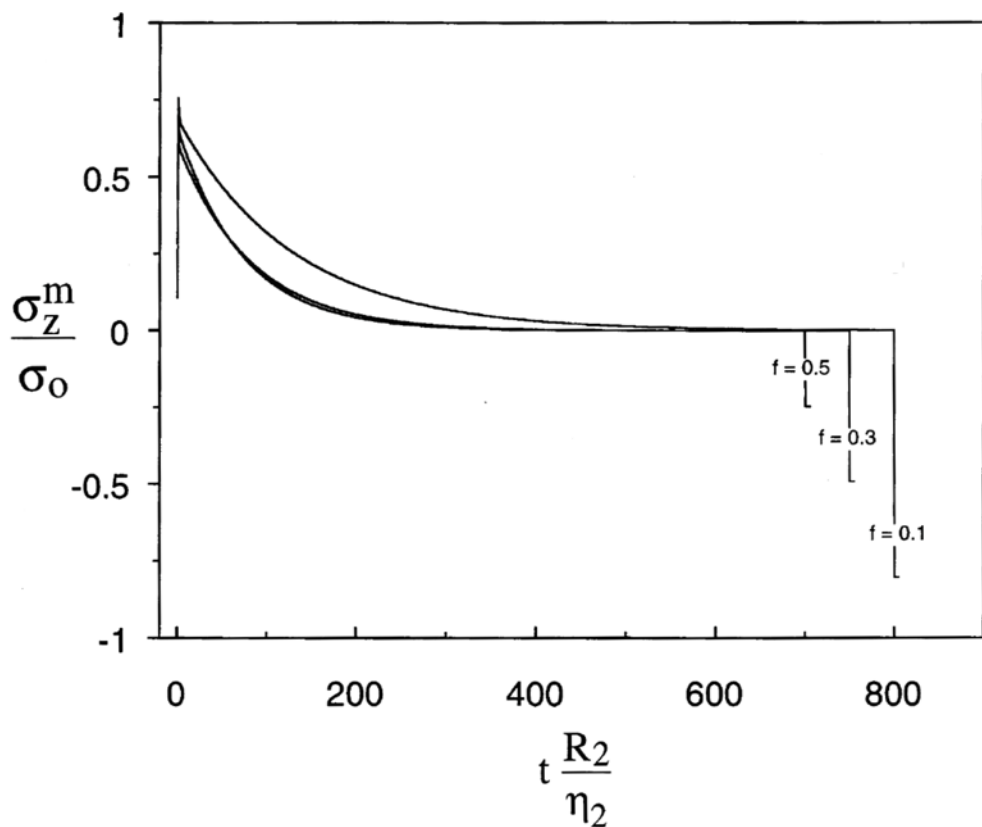


# Creep Testing Results

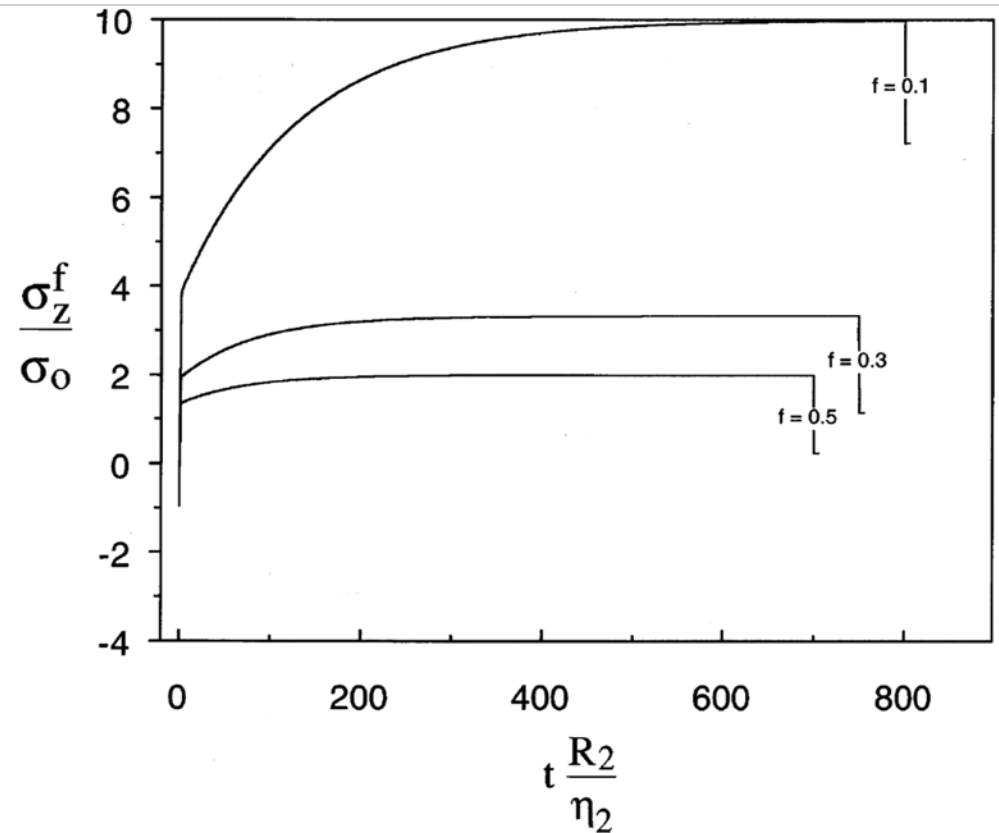


# Stress Redistribution

Stress evolution in Ni-YSZ  
(Sandwich Core)



Stress evolution in YSZ  
(Sandwich Faces)



# Summary

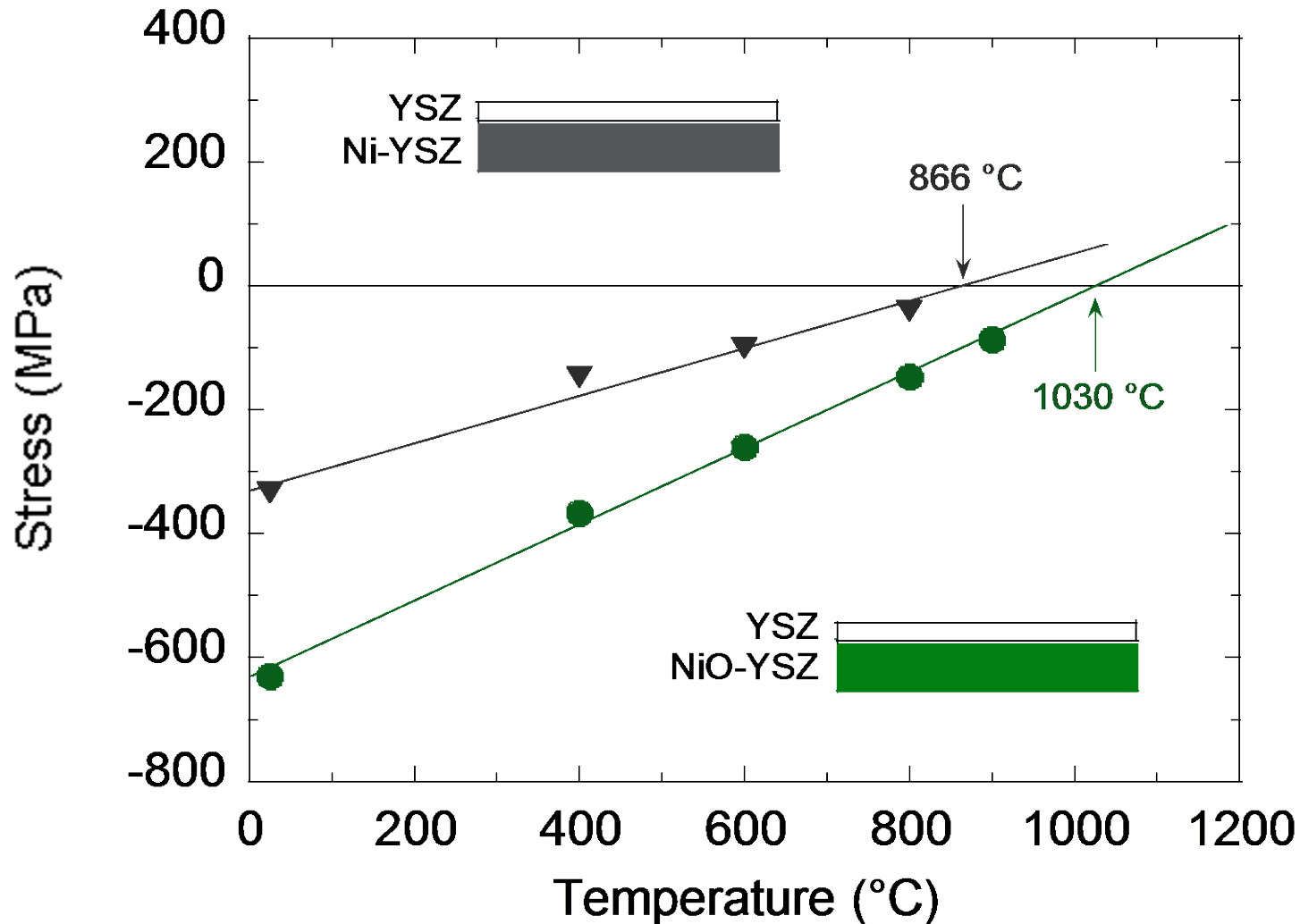
---

- The effect of creep deformation on the microstructure and functionality of anode materials is being investigated.
- The redistribution of stresses in SOFCs as a result of anode creep deformation is being investigated by measuring the evolution of residual stresses in the YSZ layers using a sandwich specimen configuration
- These results will be analyzed in the context of SOFC durability and reliability

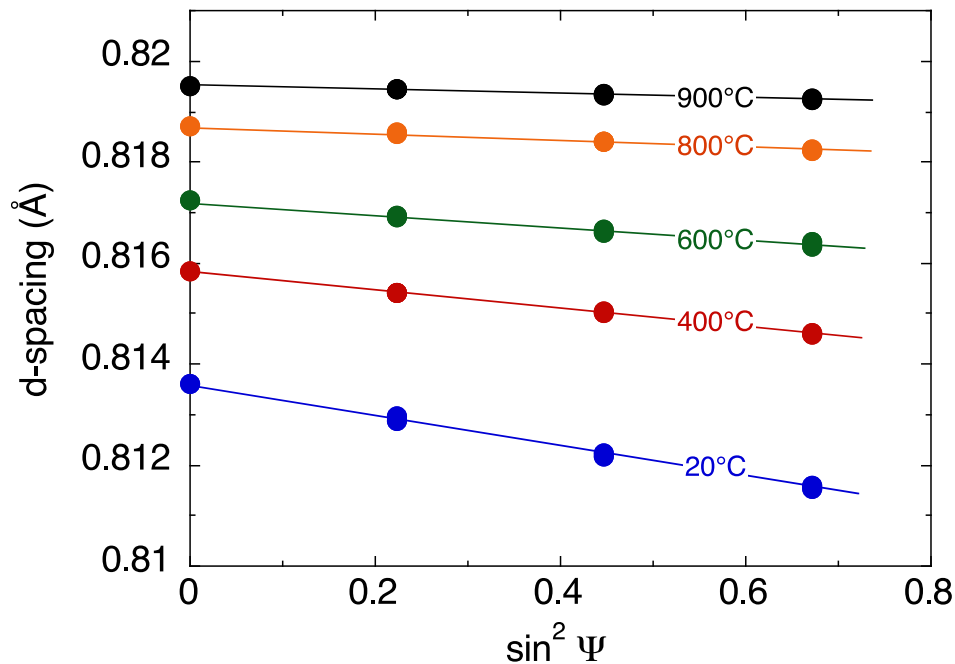
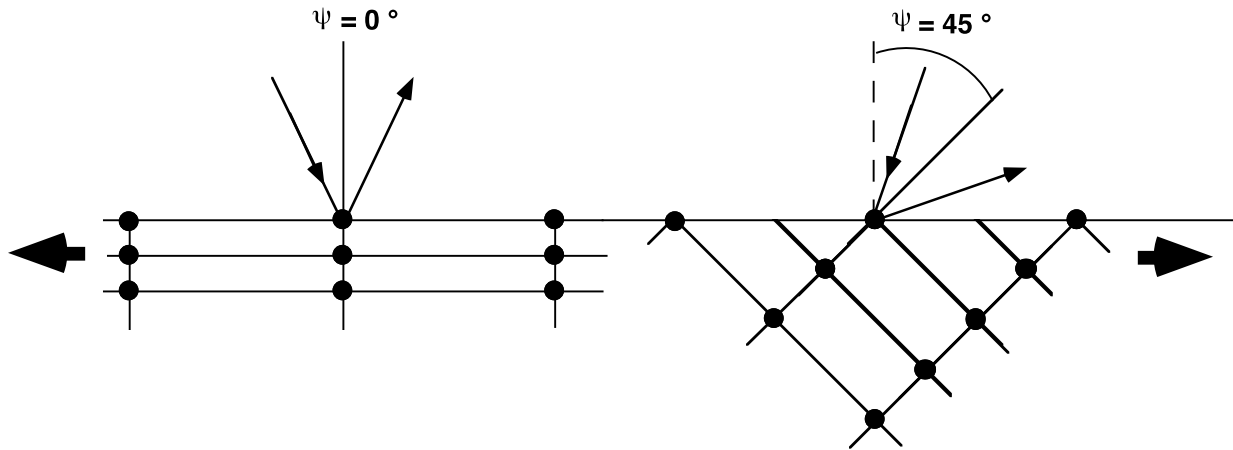


# Residual and "Reduction" Stresses (X-ray diffraction)

800°C Reduction in 4%H<sub>2</sub>-96%Ar



# Residual Stresses in Ni-YSZ Sandwich Specimens



- Sample tilting is required for accurate strain measurement with x-rays
- Peak position as a function of tilt angle,  $\psi$
- Slope of  $d$  (interplanar spacing) vs.  $\sin^2\psi$  is used to calculate strain.