



Accelerated irradiation creep testing of structural materials for advanced reactors

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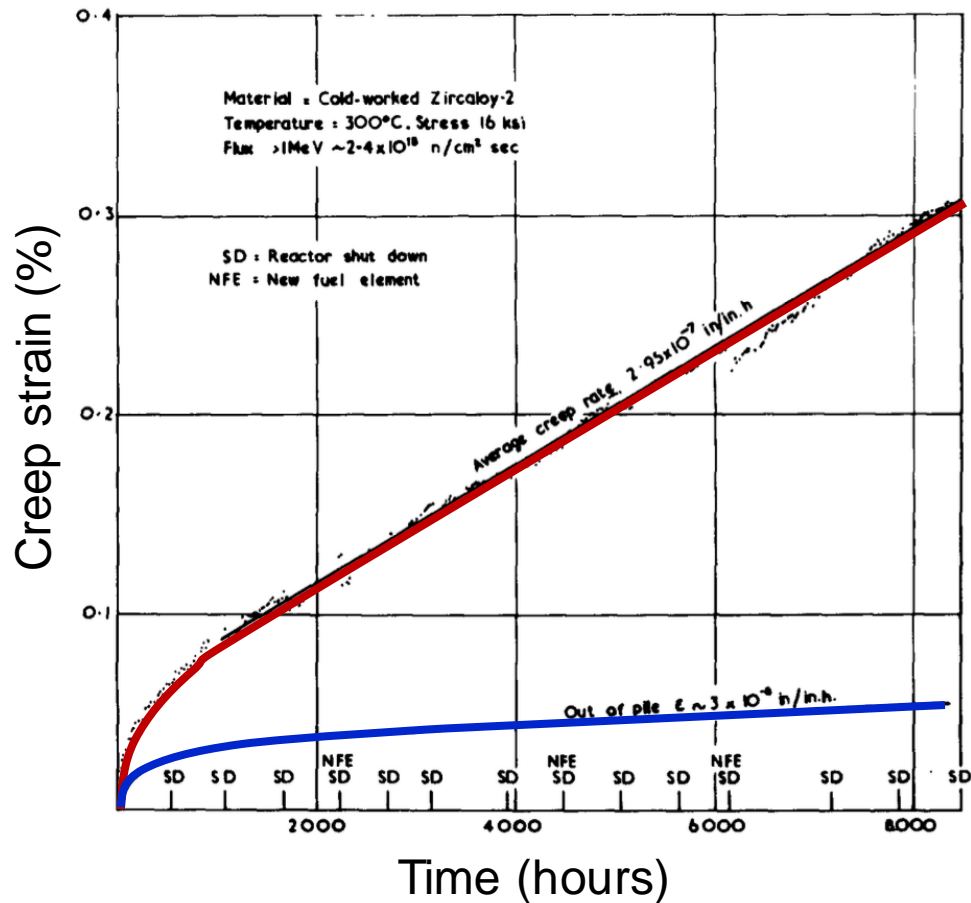
² University of Wisconsin-Madison, USA



*2024 NSUF Users Organization Annual Meeting
In-situ Irradiation, Characterization, Mechanical Testing, & Corrosion*



Irradiation can increase creep rates significantly.

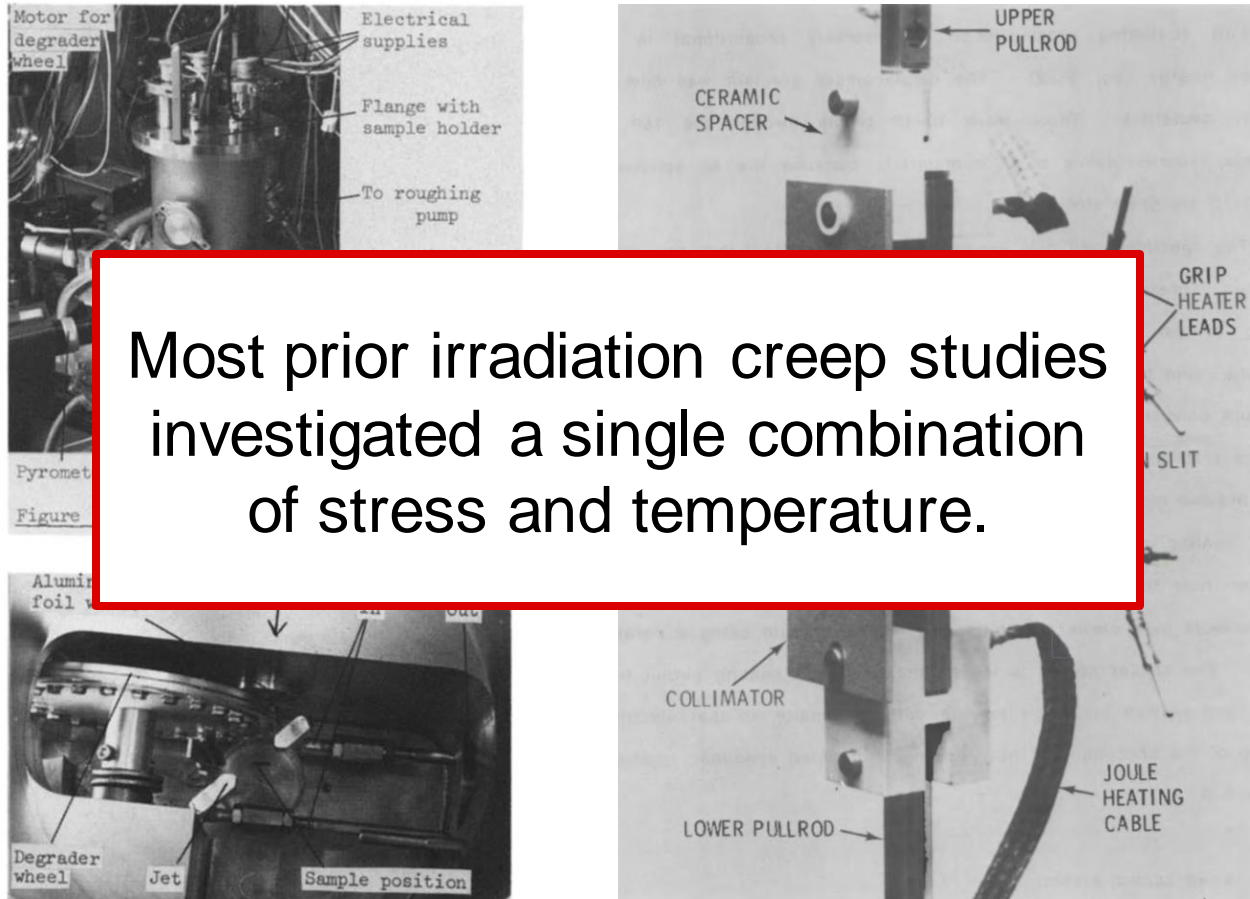


Irradiation creep
 $\dot{\epsilon} = 8.2 \times 10^{-11} \text{ s}^{-1}$

↑
One order
of magnitude!

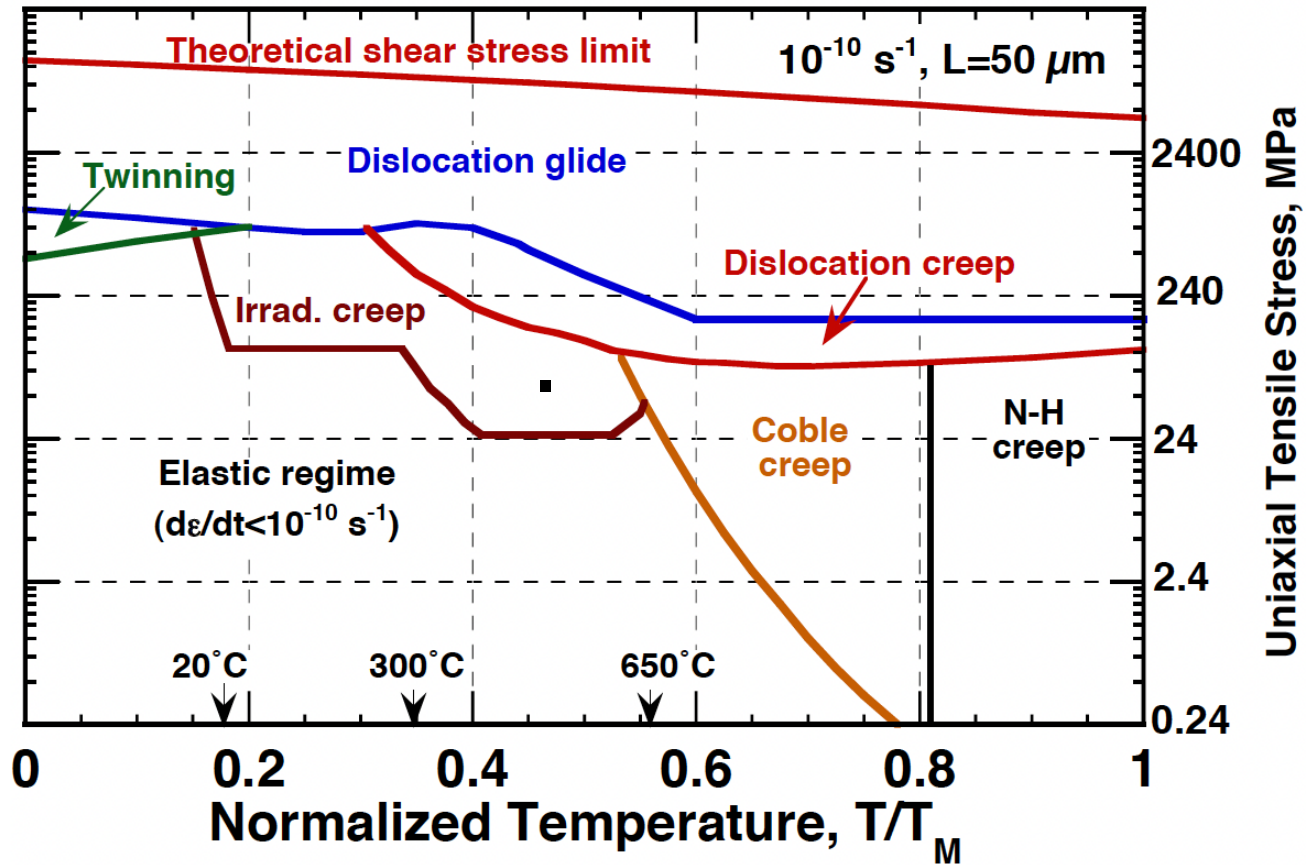
Thermal creep
 $\dot{\epsilon} = 8.3 \times 10^{-12} \text{ s}^{-1}$

Ion-irradiation creep facilities can accelerate testing.

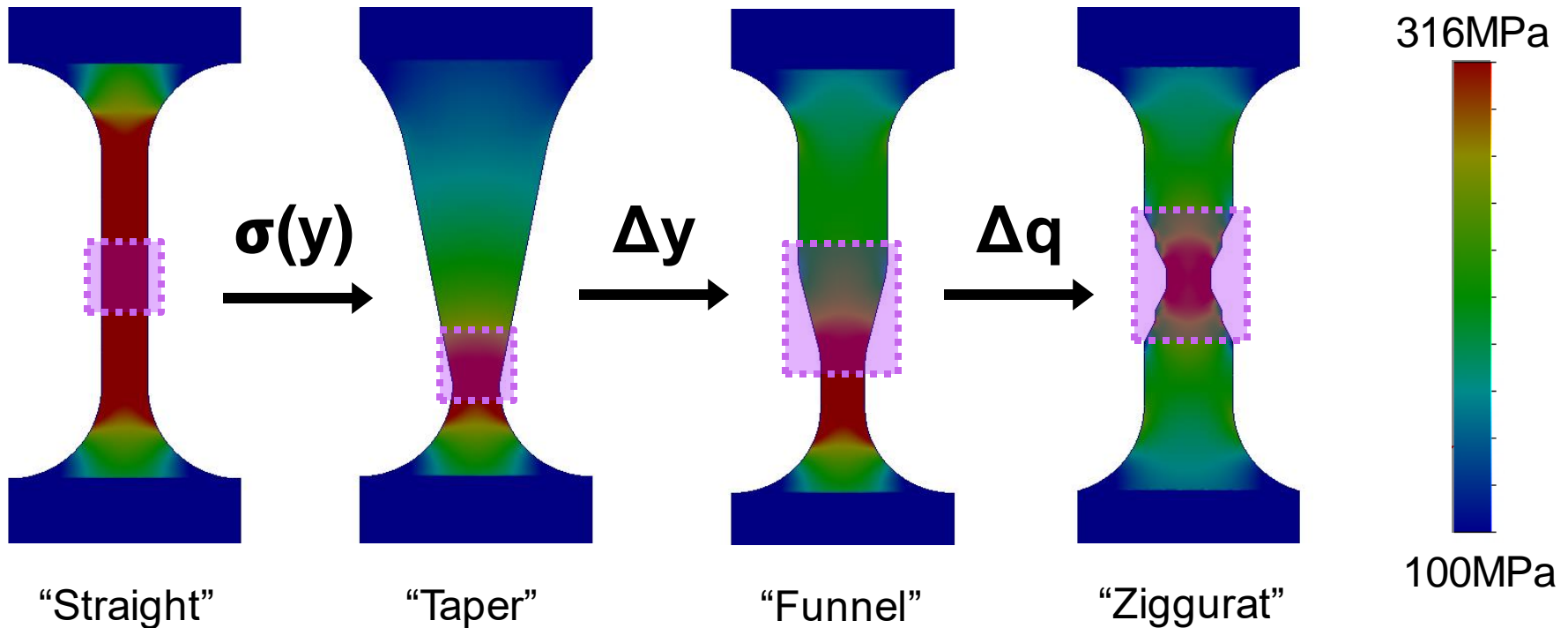


0D experiments are slow to explore parameter space.

Creep deformation mechanism map for 316 stainless steel.



Tapered specimens create multiple stress regions in a single ion-irradiation experiment.



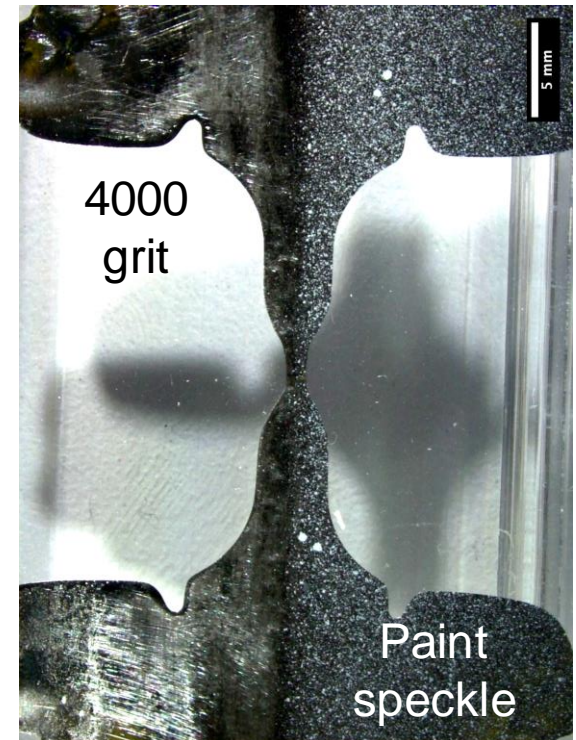
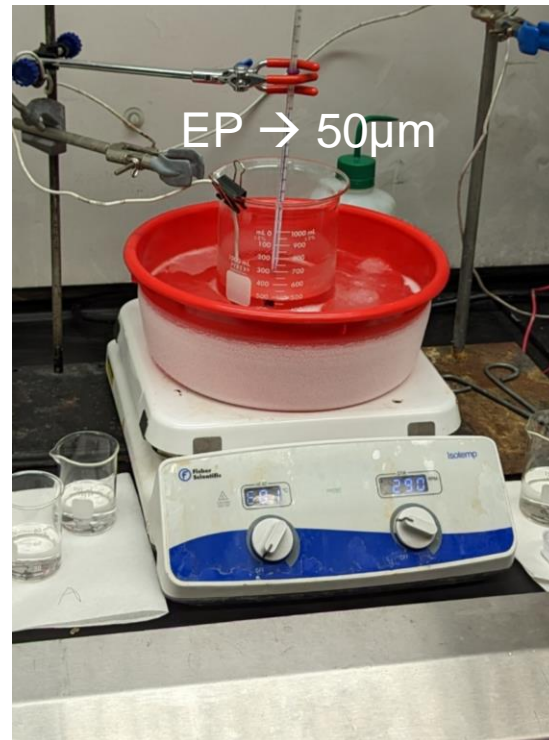
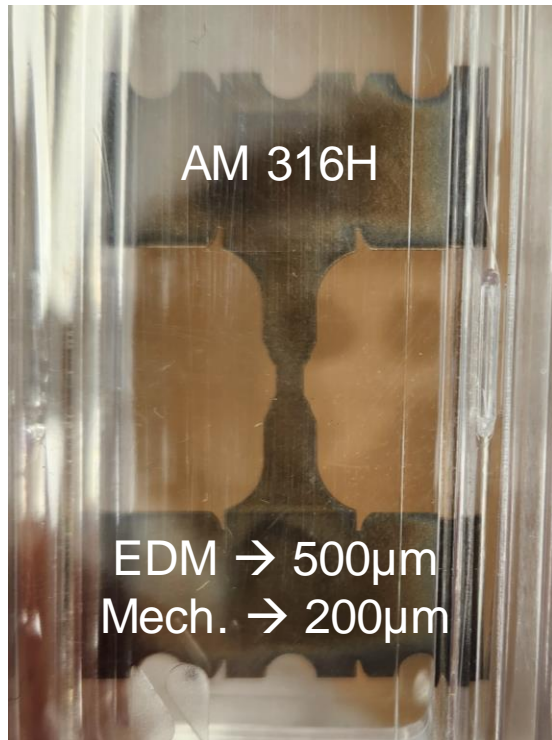
Material: 25 μm -thick annealed 316 SS foil

Stress: 158-316 MPa, **Temperature:** 550°C

3 MeV H⁺
irradiation
area

Credit: Wyatt Peterson

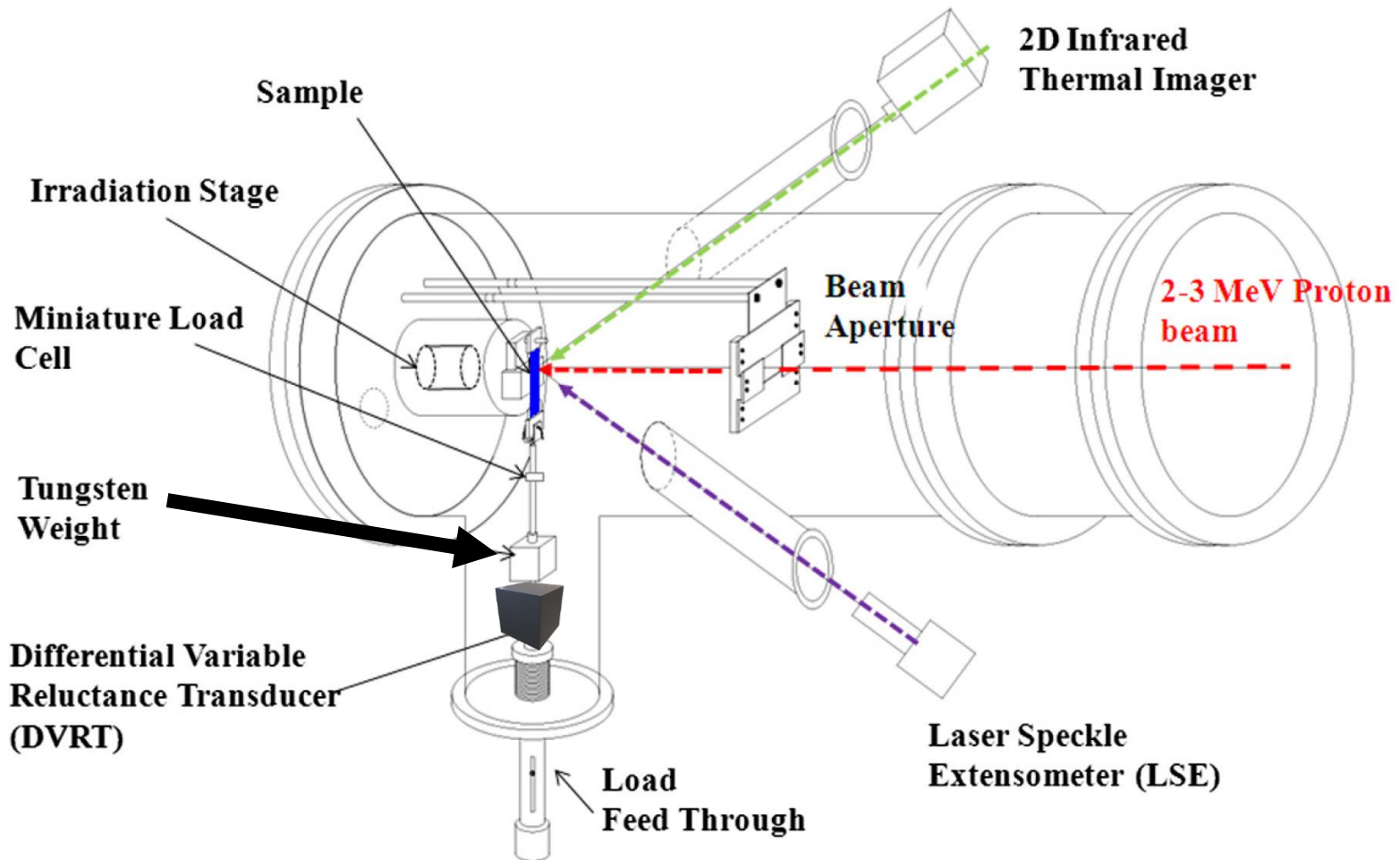
Bulk samples are prepared using EDM,
followed by mechanical- and electro- polishing.



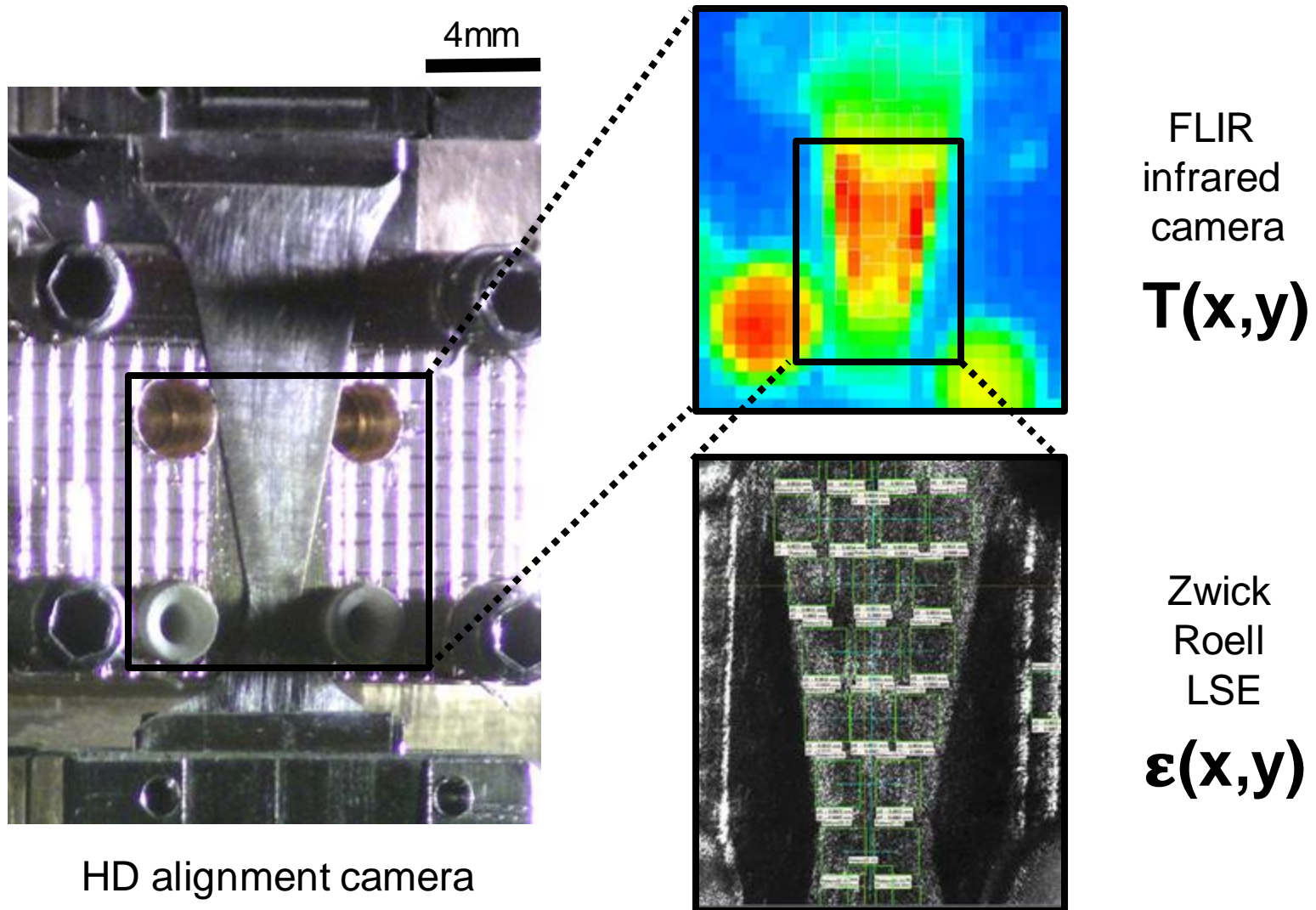
RTE #4654 – ‘Quantifying the effect of simultaneous vs. sequential irradiation on creep performance of additively manufactured austenitic stainless steel’ (PI Massey).

RTE #4817 – ‘Investigating the evolution of M₂₃C₆ and MX-type precipitates in additively manufactured Grade 91 steel under high T. simultaneous & sequential stress & irradiation’ (PI Narra)

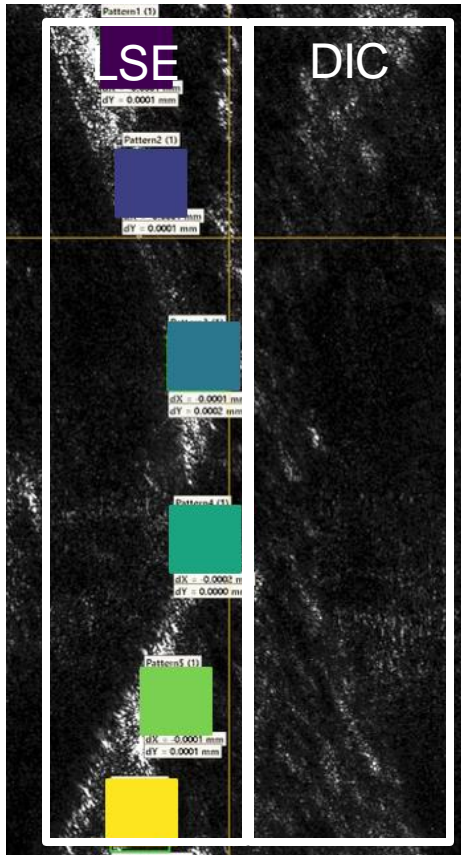
The current MIBL irradiation creep stage uses W deadweights to apply a constant load.



Imaging captures local temperature & strain fields.

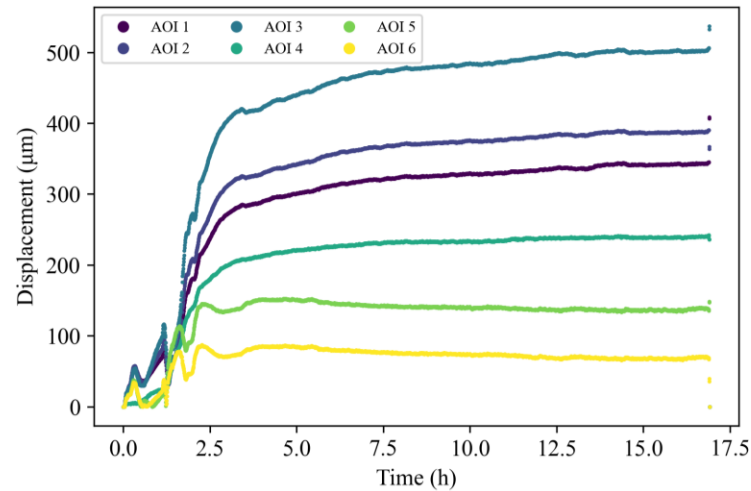


Multiple non-contact extensometry techniques are being evaluated to extract strain at each position.



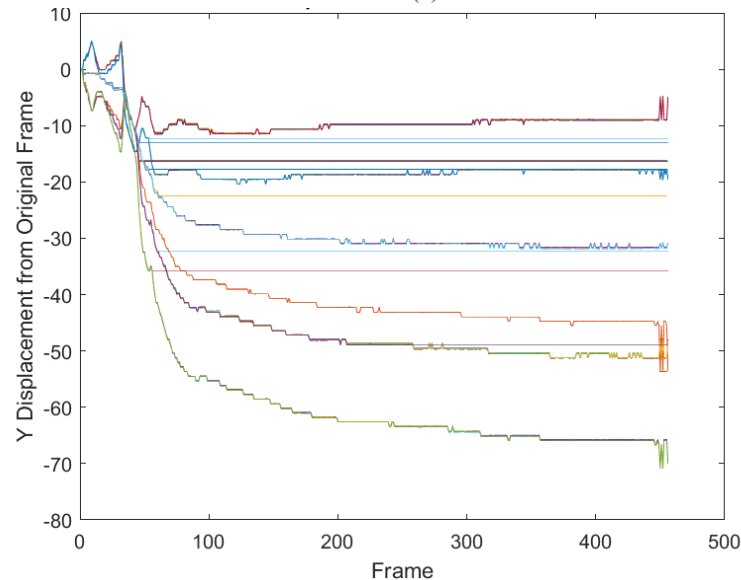
"Ziggurat" sample

Credit: Mackenzie Warwick & Ben Arms



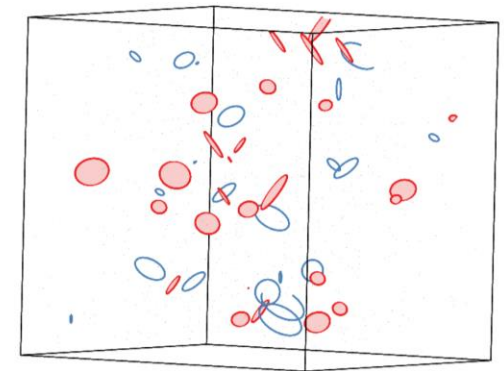
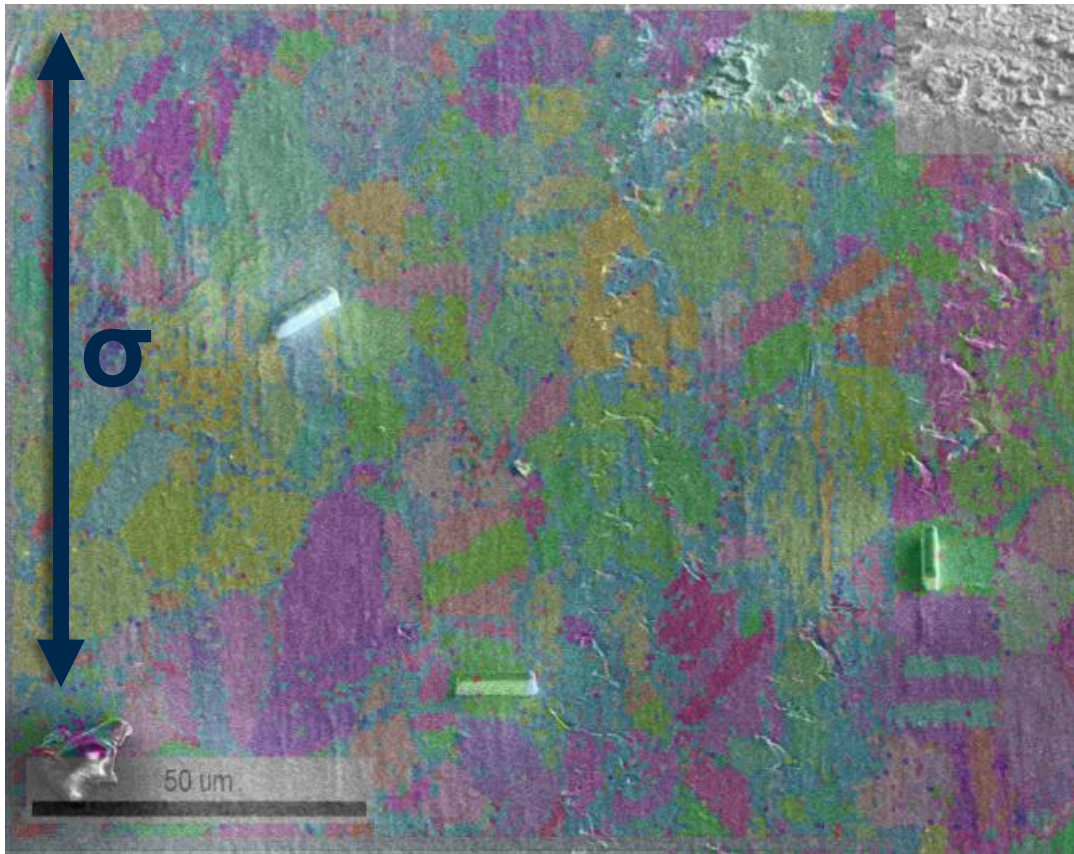
Laser Speckle Extensometer (LSE) Displacement





≈



Digital Image Correlation (DIC) Displacement

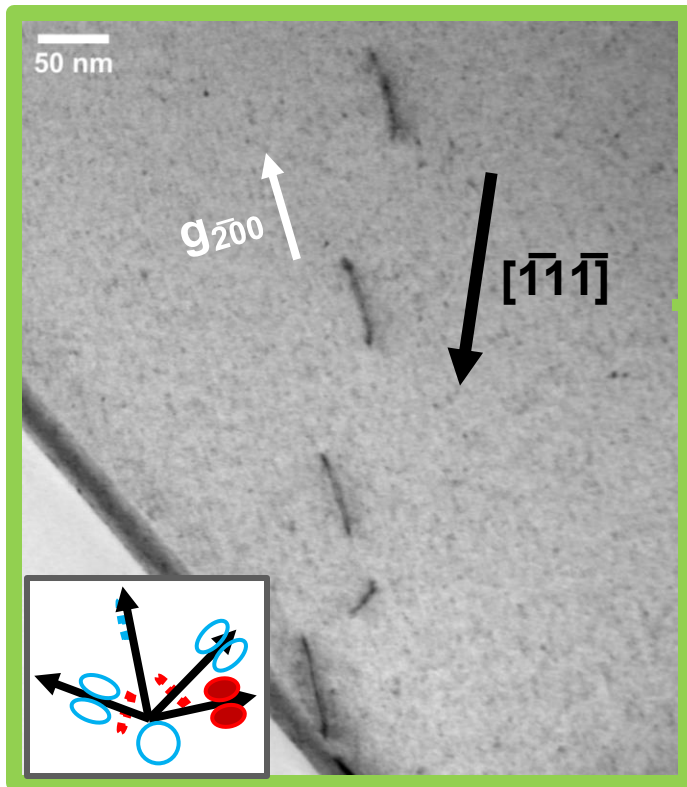
FIB lift outs are used to characterize microstructural evolution: relationship to applied stress is important.



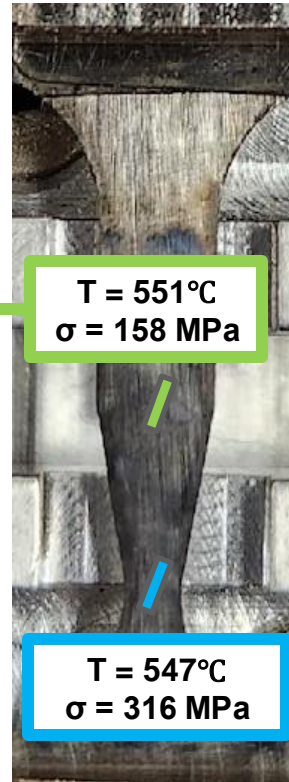
-  Perfect loops
-  Edge-on perfect loops
-  Faulted loops
-  Edge-on faulted loops

Initial characterization shows distinct microstructures.

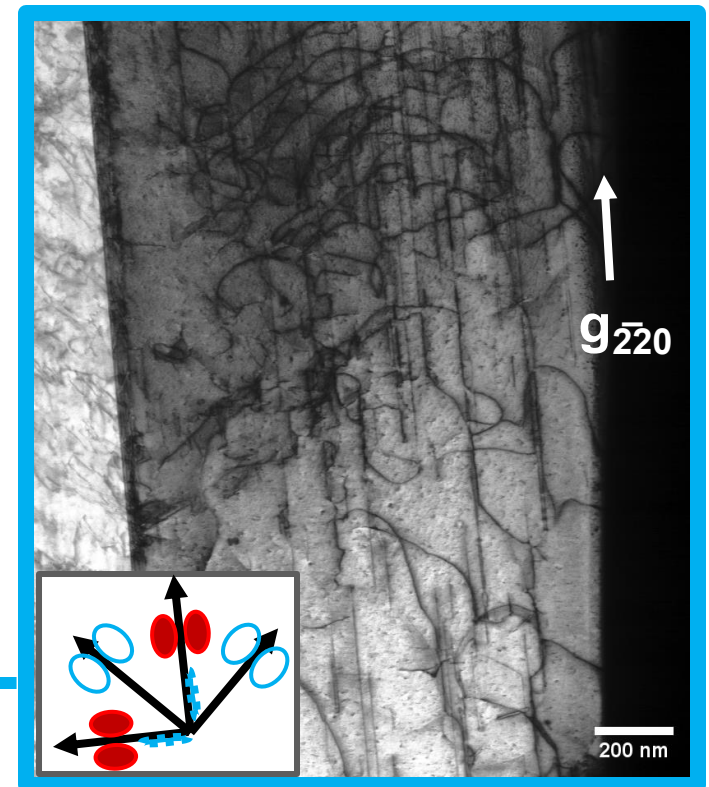
158 MPa



On-Zone S/TEM (011)
Average length: 59.3 ± 7.1 nm



316 MPa

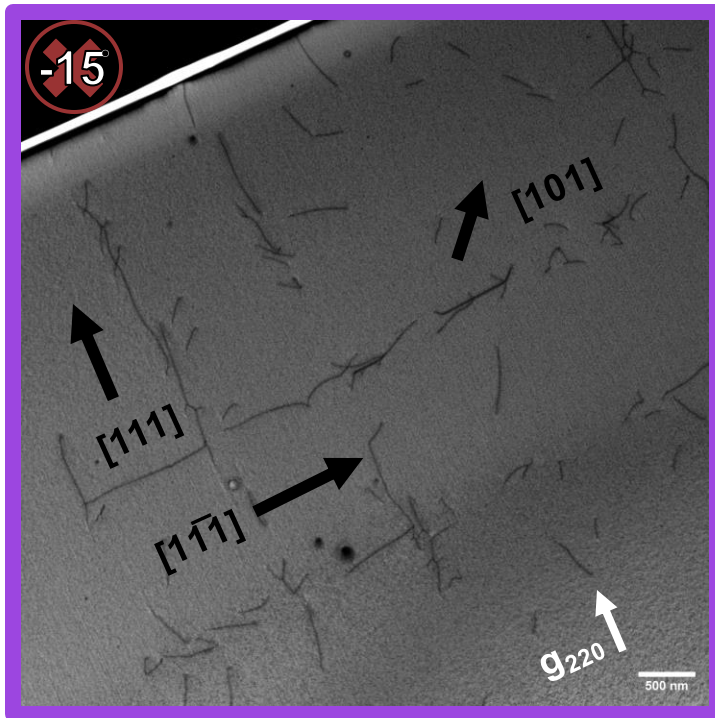


On-Zone S/TEM (001)
Dislocation Network Density: $3 \times 10^{14} \text{ m}^{-2}$

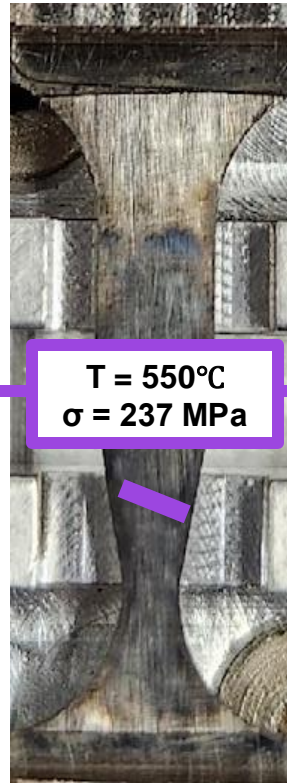
Credit: Mackenzie Warwick

Further TEM characterization is in progress...

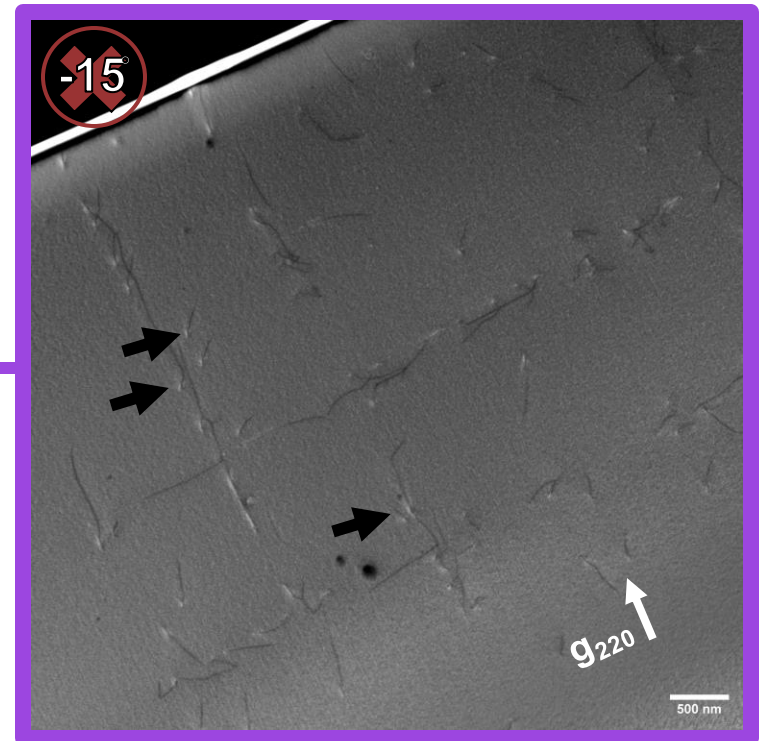
237 MPa



On-zone S/TEM BF <001>



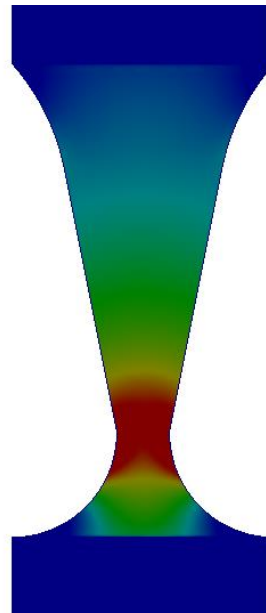
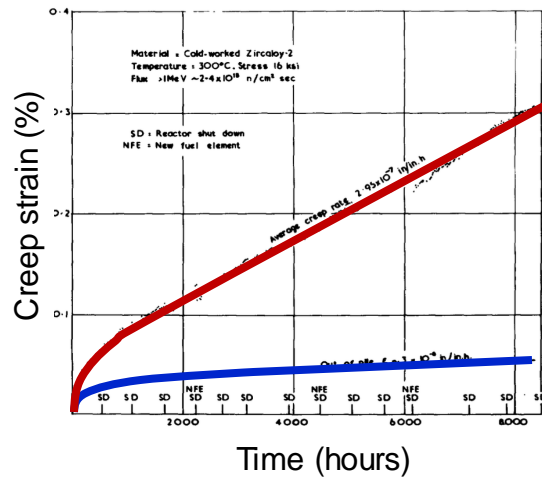
237 MPa



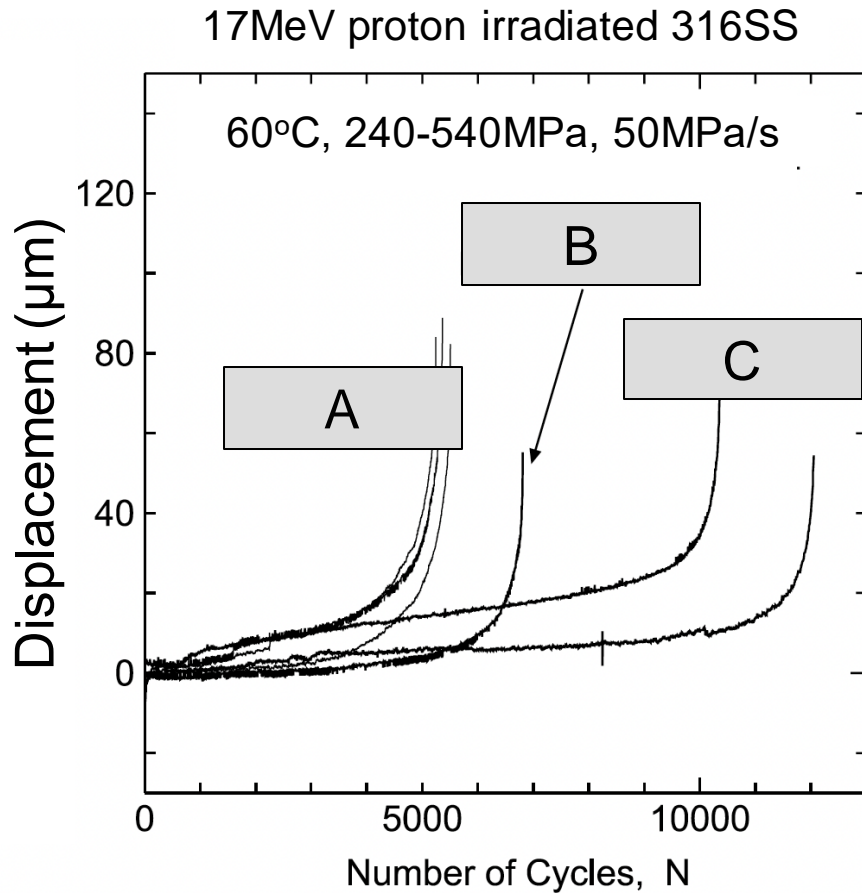
On-zone S/TEM DF <001>

Now characterization has become the bottleneck!

Accelerated irradiation creep testing of structural materials for advanced reactors



Nuclear materials' properties depend on the precise combination of irradiation, temperature, and load.



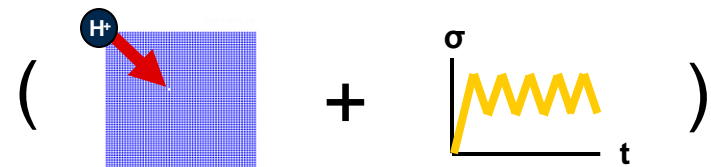
1. Unirradiated.



2. Tested post-irradiation.



3. Tested under irradiation.



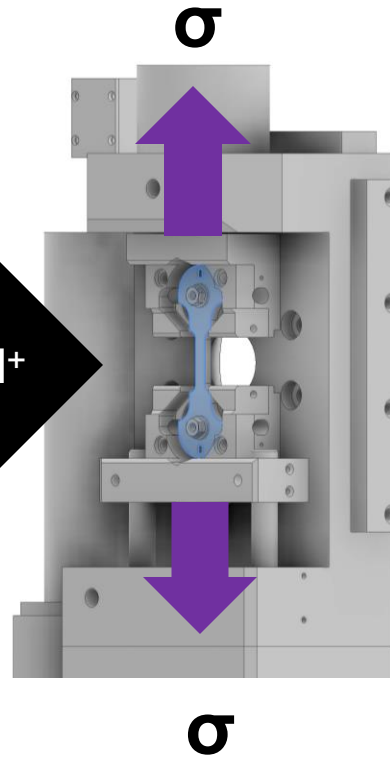
Simultaneous ion-irradiation creep fatigue (ICF) will be investigated at the Michigan Ion Beam Lab*

NewTec MT1000
in-situ tensile stage

Grip heating up to 1000°C

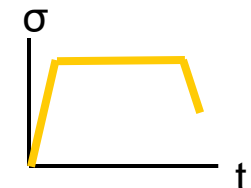
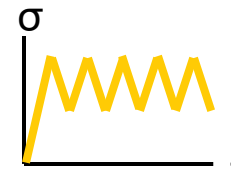
IRRADIATION 3 MeV H⁺

in situ and
in operando
tensile testing



T > 0.4T_m = CREEP

σ(t) = FATIGUE

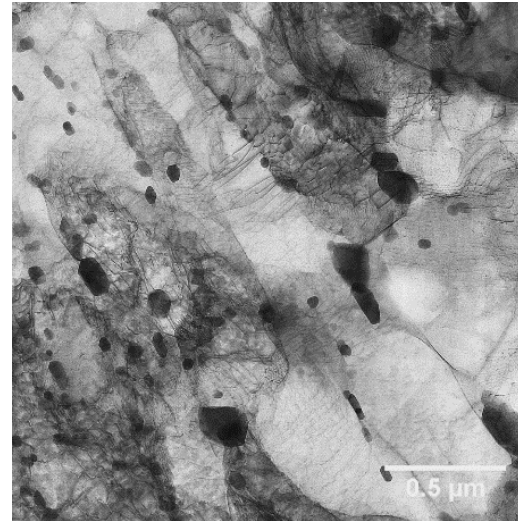
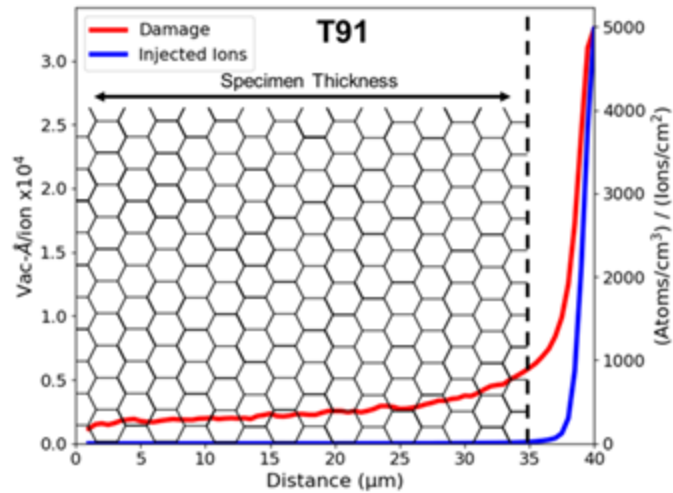


CAD courtesy of NewTec Scientific



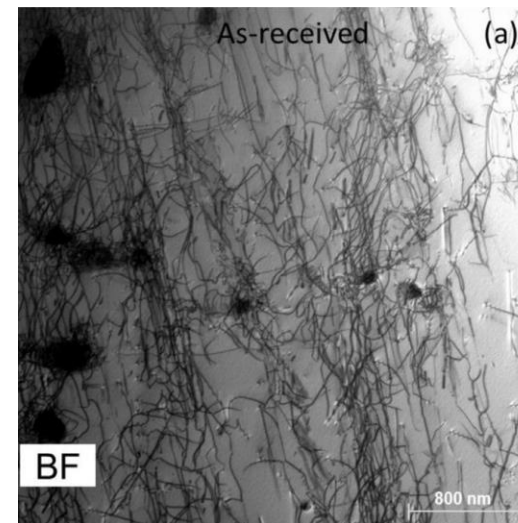
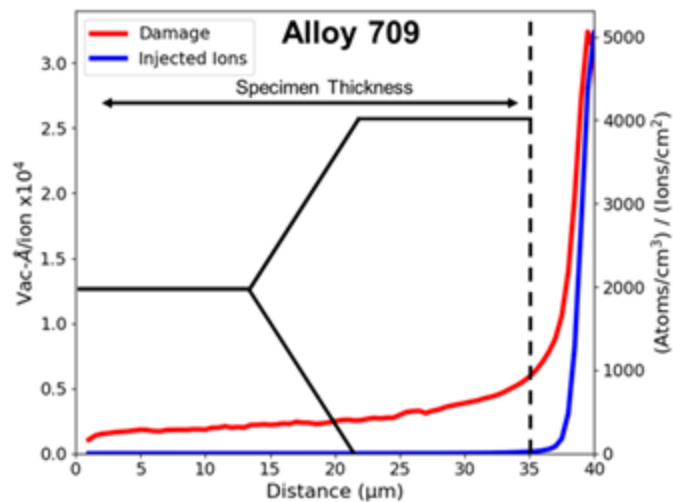
* and at the Wisconsin Ion Beam Lab!

Specimen thickness & microstructural length scale are critical parameters.



Grade 91

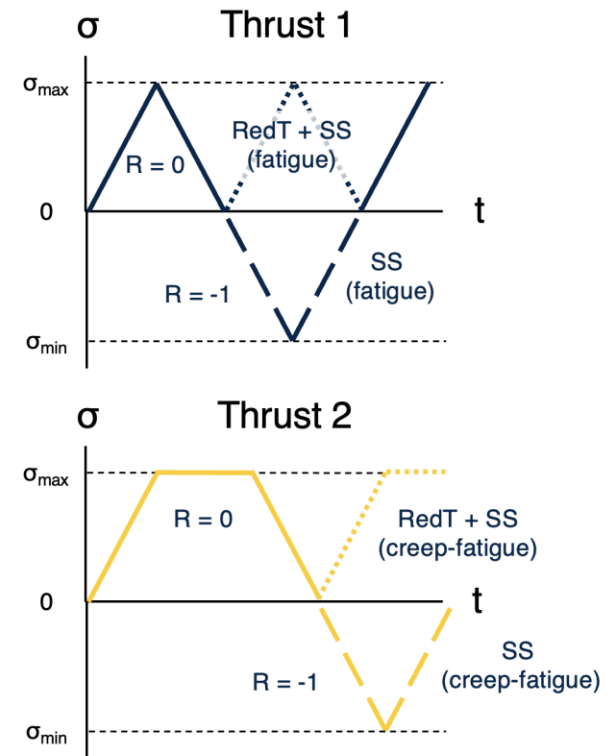
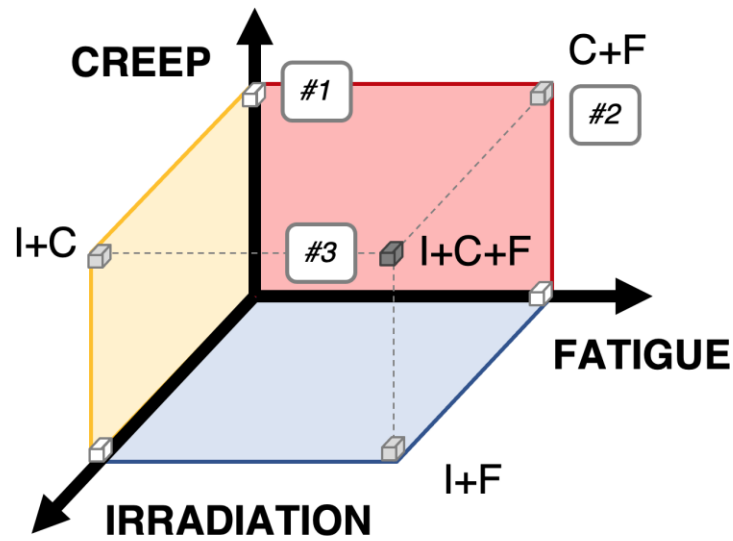
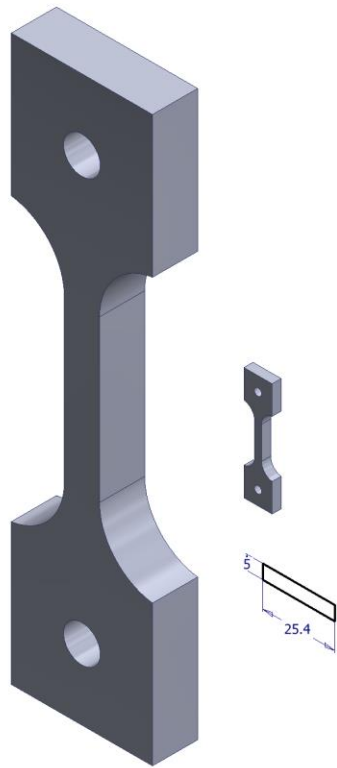
Zhang et al.,
Int. J. Fatigue,
125 (2019)
440-453



Alloy 709

Zhang et al.
J. Nucl. Mater.
553 (2021)
153052

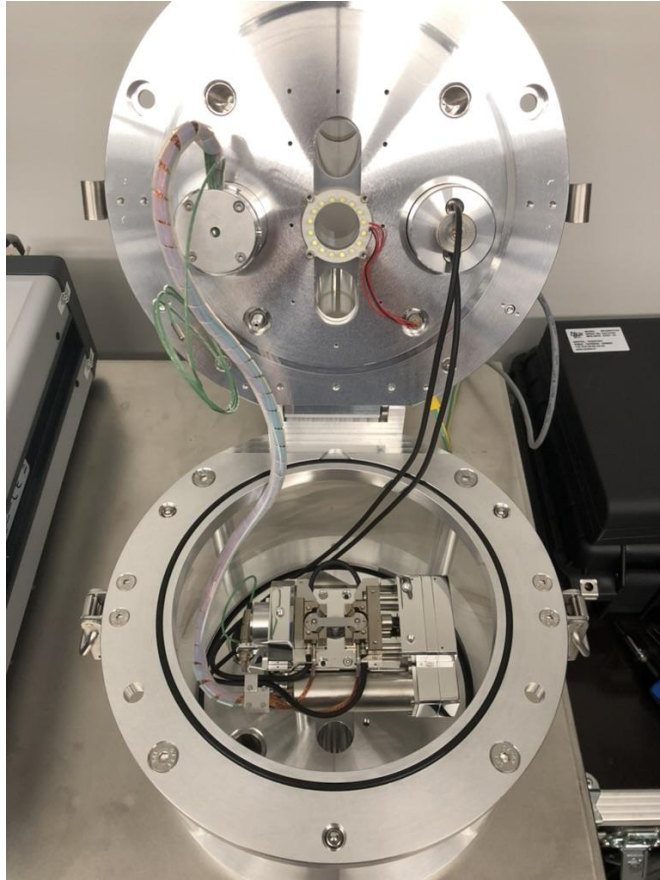
The effects of sample geometry and creep-fatigue loading waveform will be investigated.



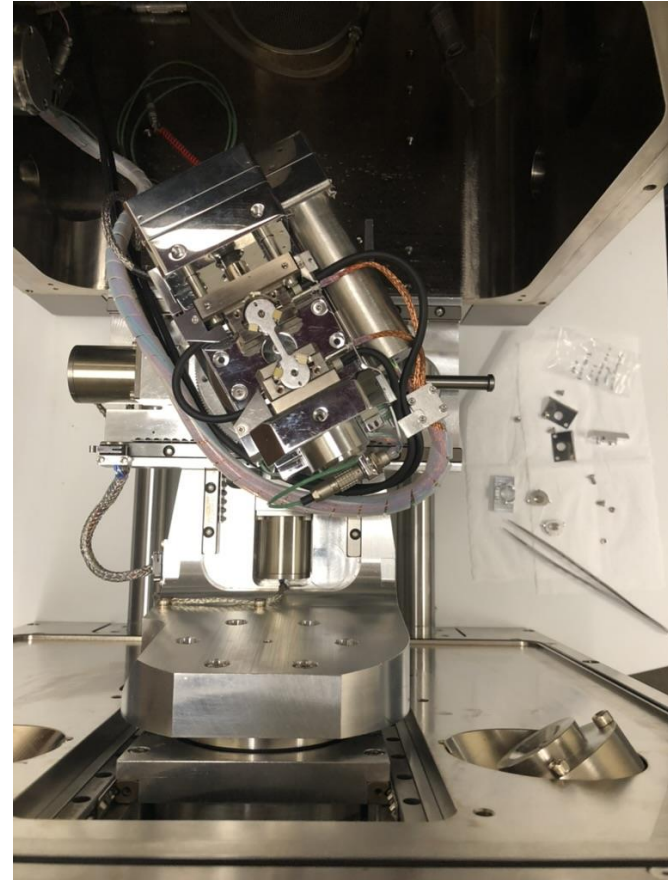
Standard size (SS)
Reduced-thickness (RedT)

Stress intensity ratio (R)
Hold time under load.

Miniature tensile rig received and tested within SEM.

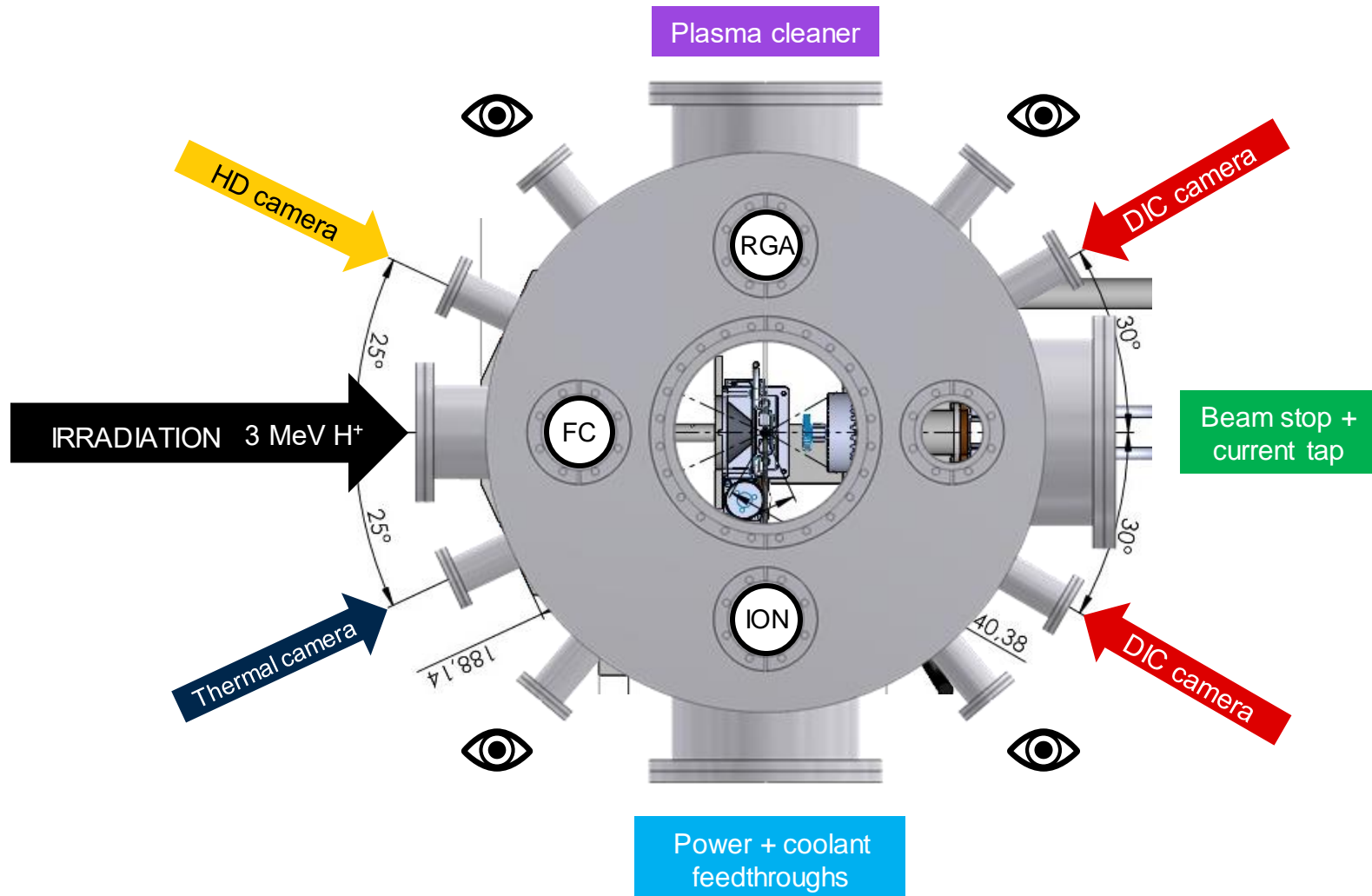


MT1000 within exoSEM



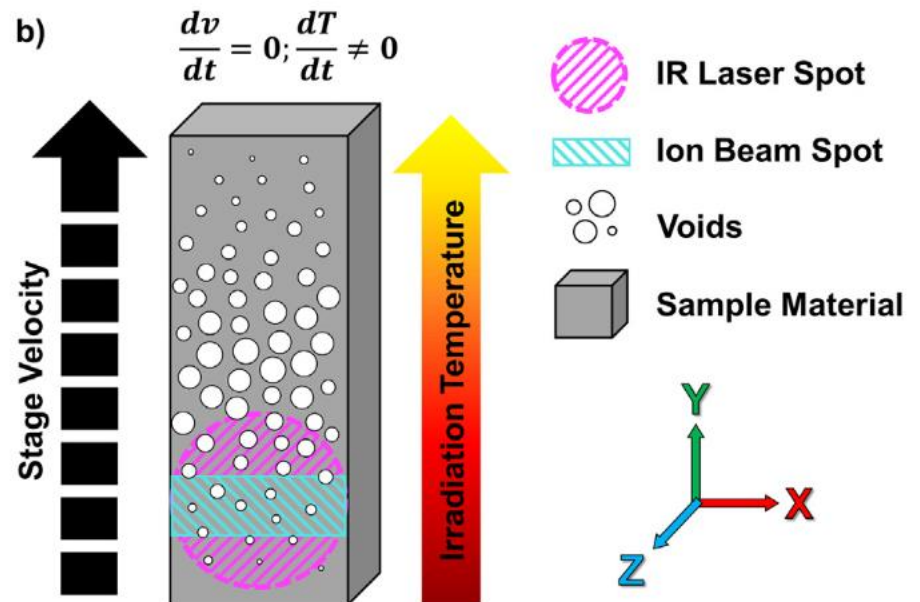
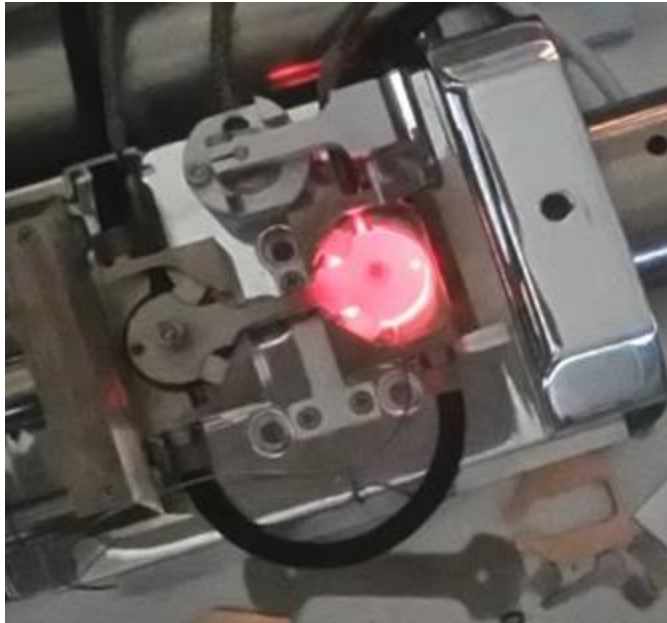
within TESCAN MIRA3

Beamline chamber in the process of being designed.



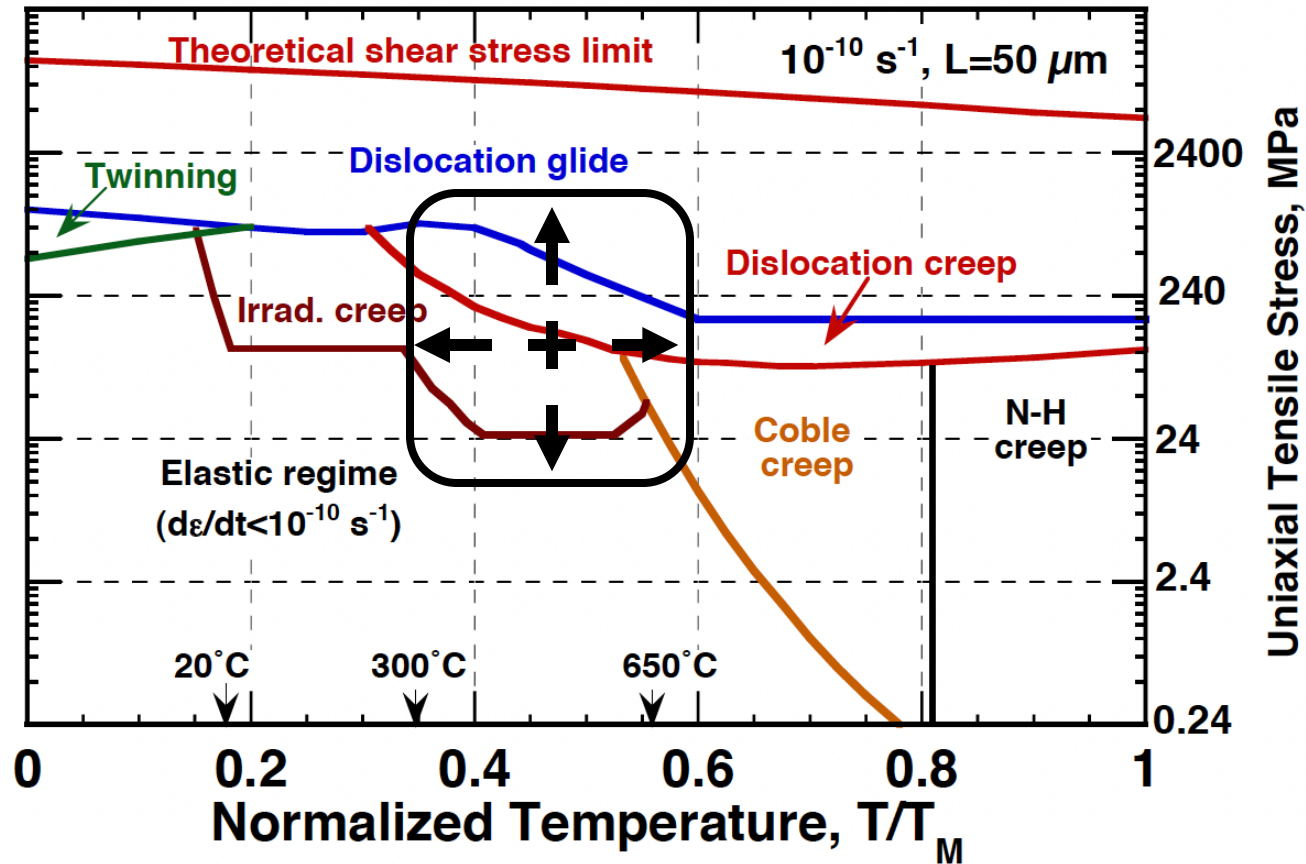
Drawing courtesy of NewTec Scientific

Independent grip heaters allow for the creation of temperature gradients along the sample length.

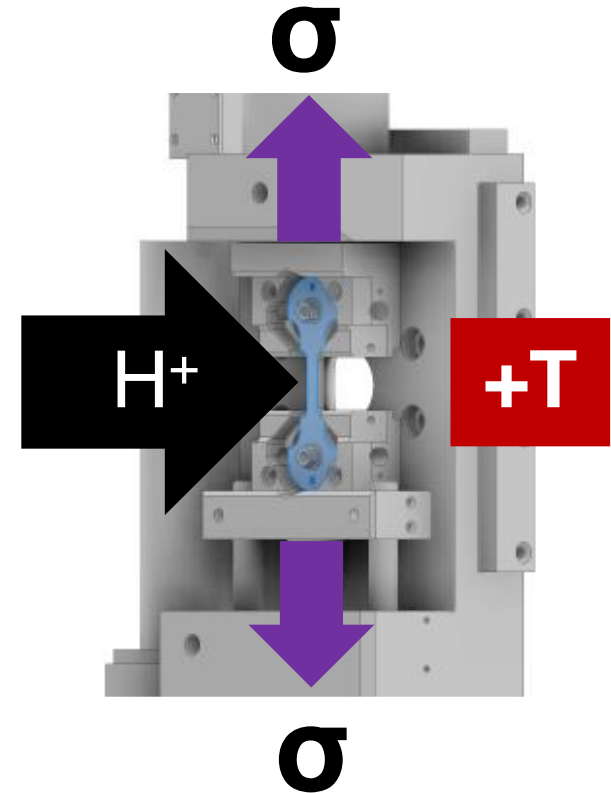
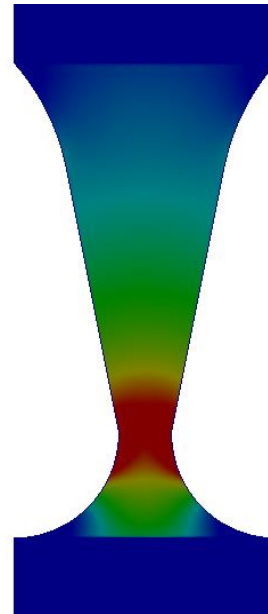
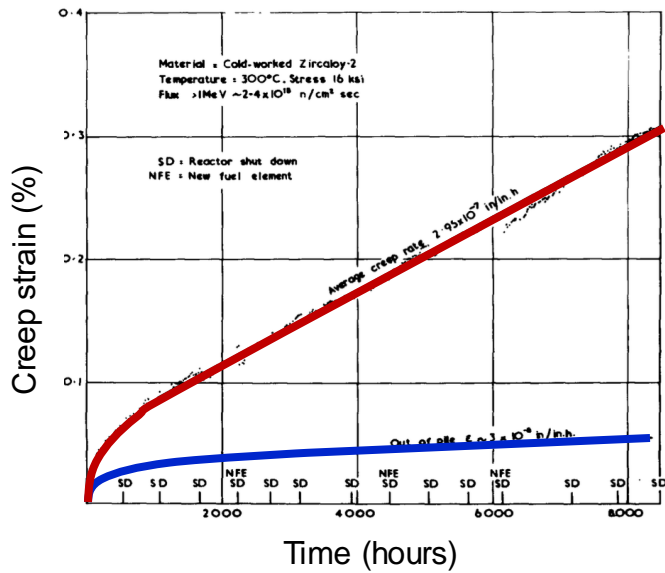


1D gradient experiments will accelerate exploration.

Creep deformation mechanism map for 316 stainless steel.



Accelerated irradiation creep testing of structural materials for advanced reactors



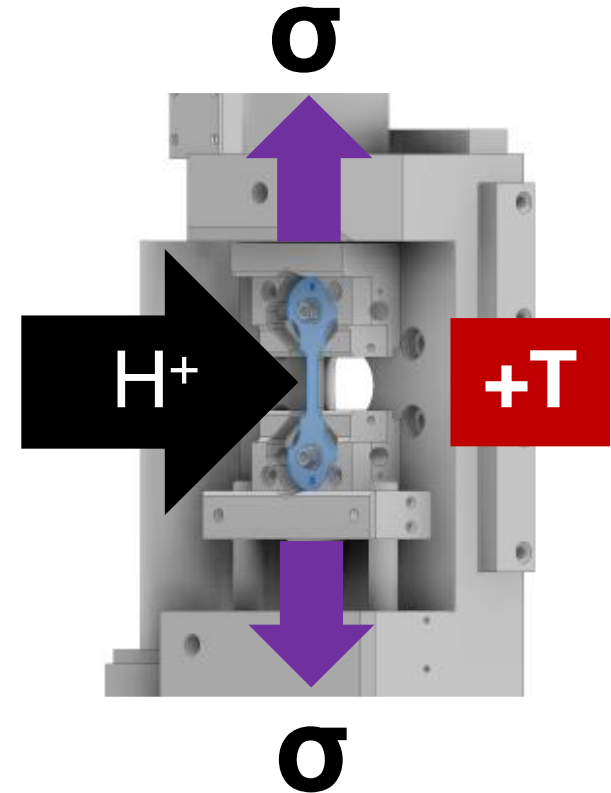
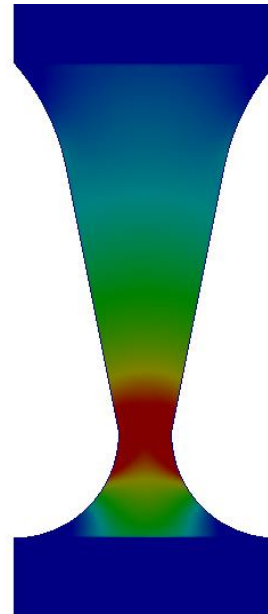
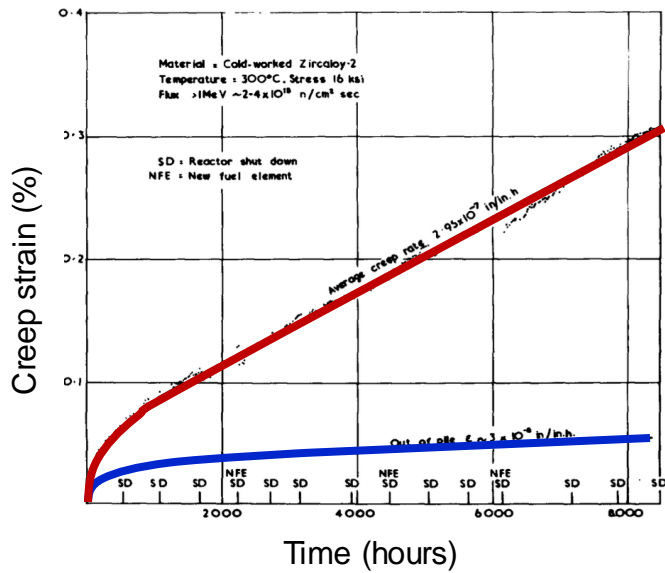
Acknowledgements



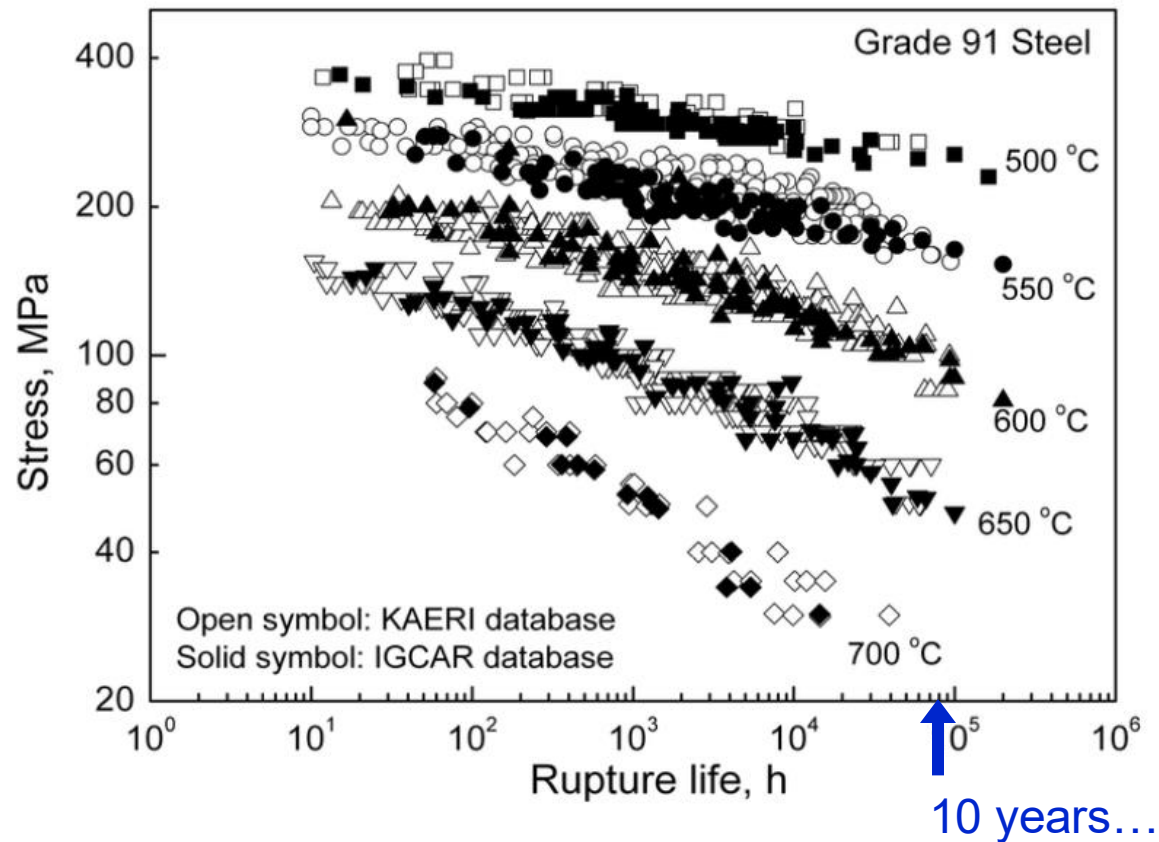
U.S. Department of Energy

1. Accelerated irradiation creep testing coupled with self-adaptive accelerated molecular dynamics simulations for scalability analysis.
2. Mechanism driven evaluations of sequential and simultaneous irradiation-creep-fatigue testing.

Accelerated irradiation creep testing of structural materials for advanced reactors

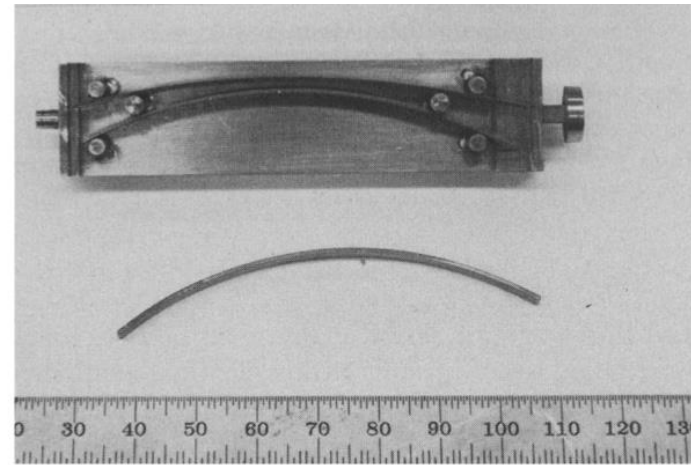
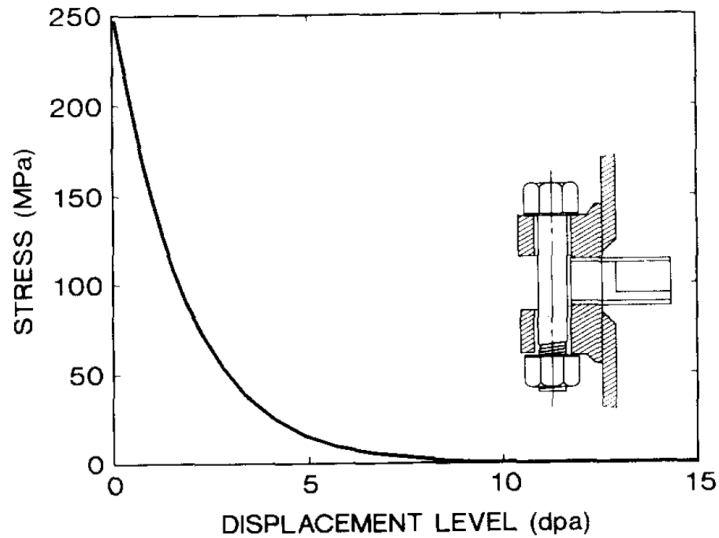


Elevated temperatures & stress cause thermal creep.



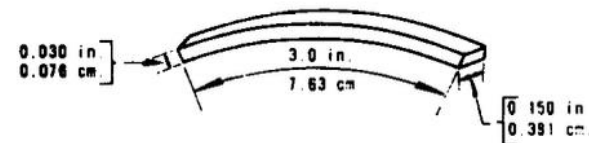
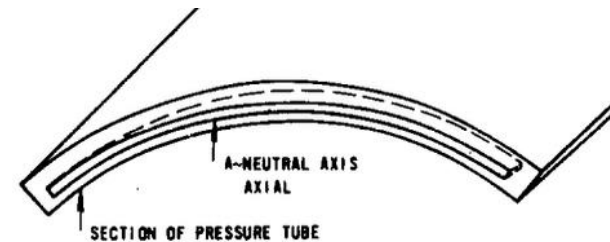
Increasing temperatures decrease the lifetime.

Stress relaxation measurements are inaccurate at long times and difficult to measure in-reactor.



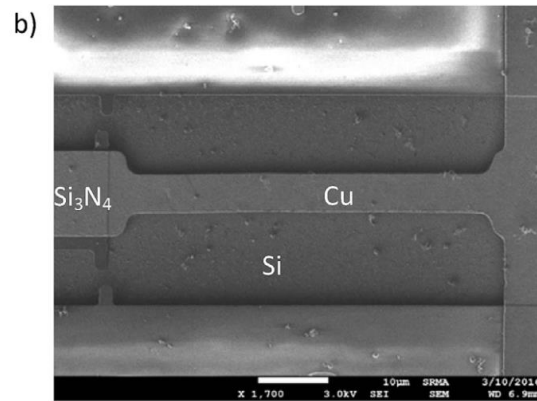
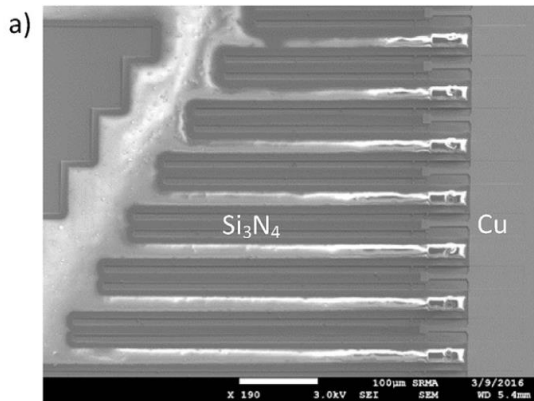
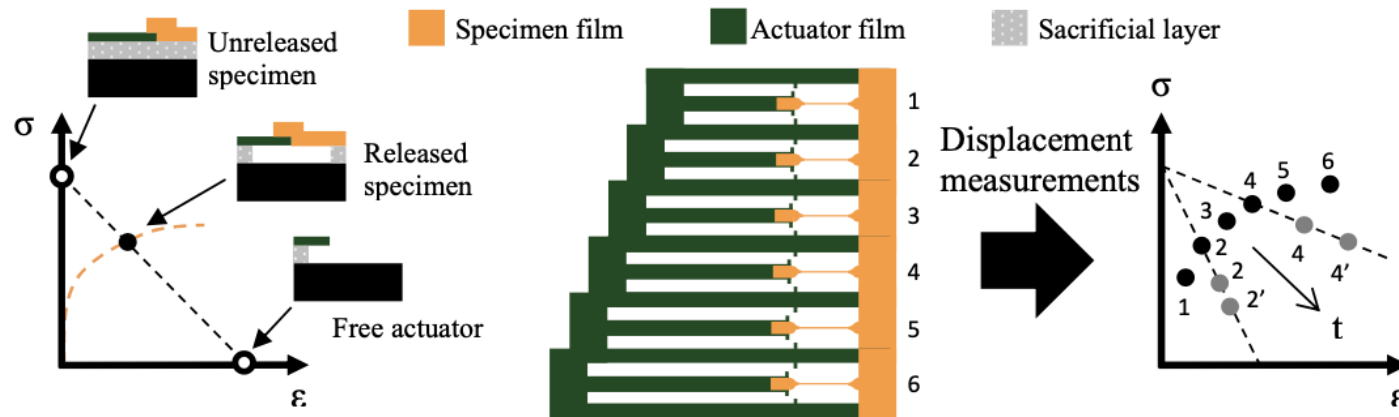
$$\dot{\epsilon} \propto \sigma(t)$$

strain rate depends on stress
which varies over time.



Grossbeck and Mansur, *J. Nucl. Mater.* 179 (1991) 130
Causey et al. *J. Nucl. Mater.* 159 (1988) 101

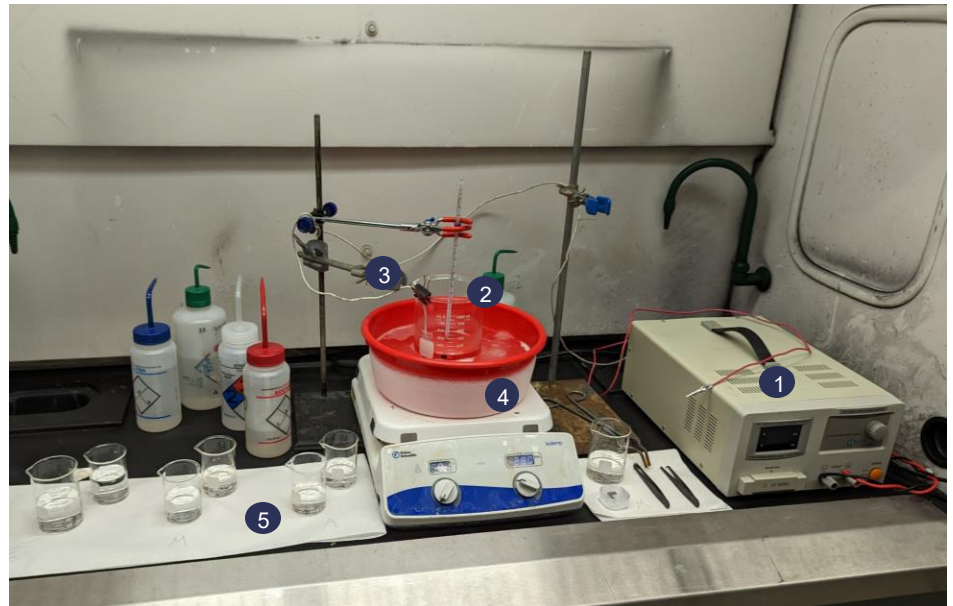
MEMS irradiation creep can perform parallel tests.



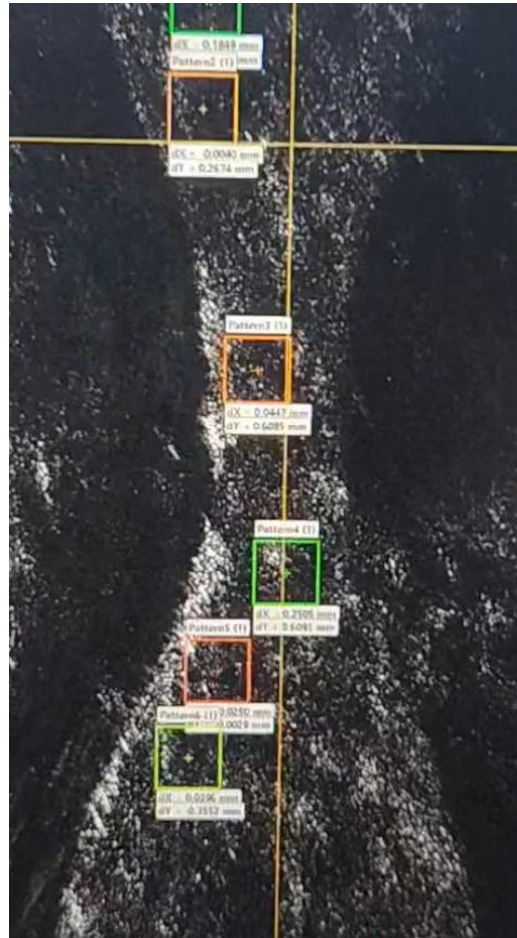
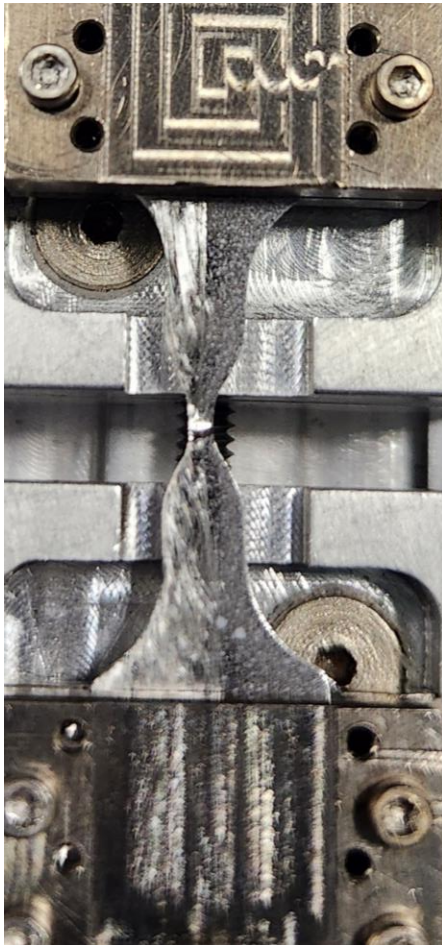
How representative is 200 nm-thick Cu to 'bulk' material creep behavior?

Electropolishing – Methodology

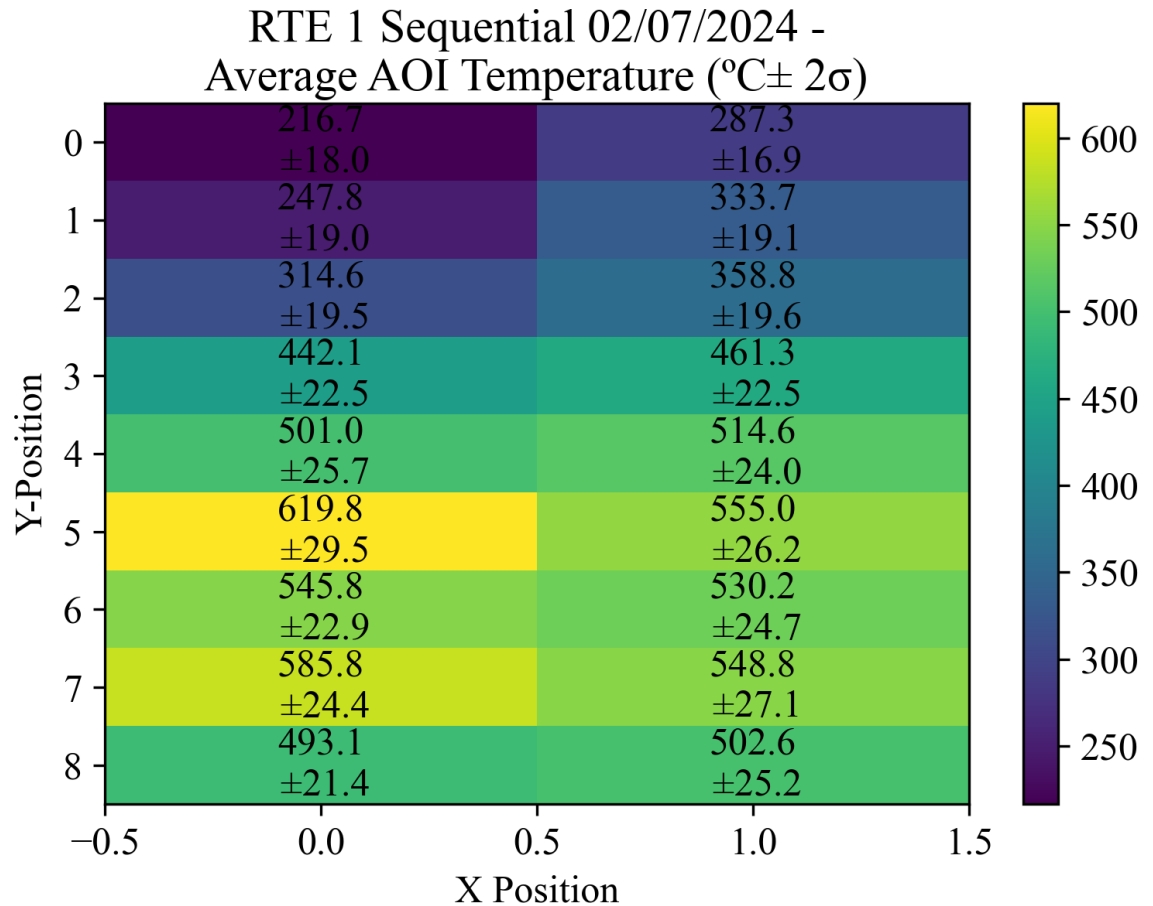
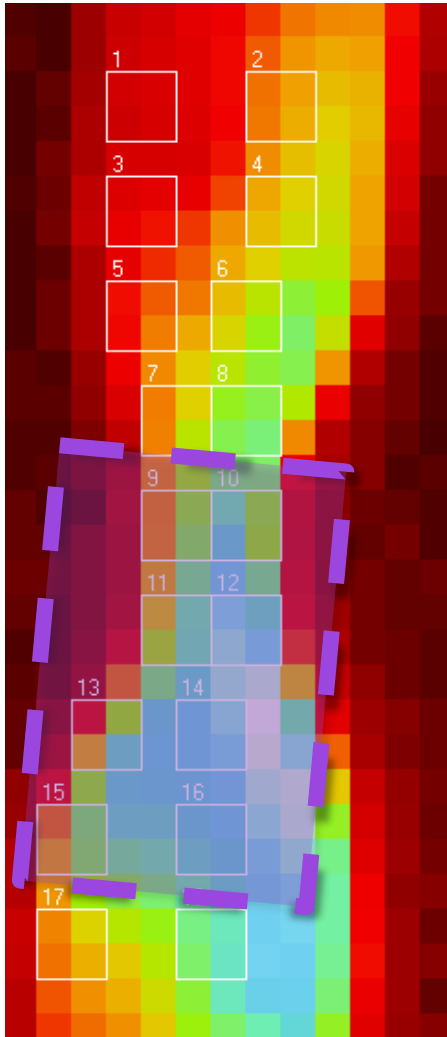
- ① Voltage source, set to 40V
- ② Acid solution; 10% Perchloric acid, 90% Methanol
- ③ Negative lead, connected to platinum mesh
- ④ Methanol bath, cool to -45 °C
- ⑤ Sample cleaning process; Acetone, Methanol, and Ethanol for 20 seconds twice each



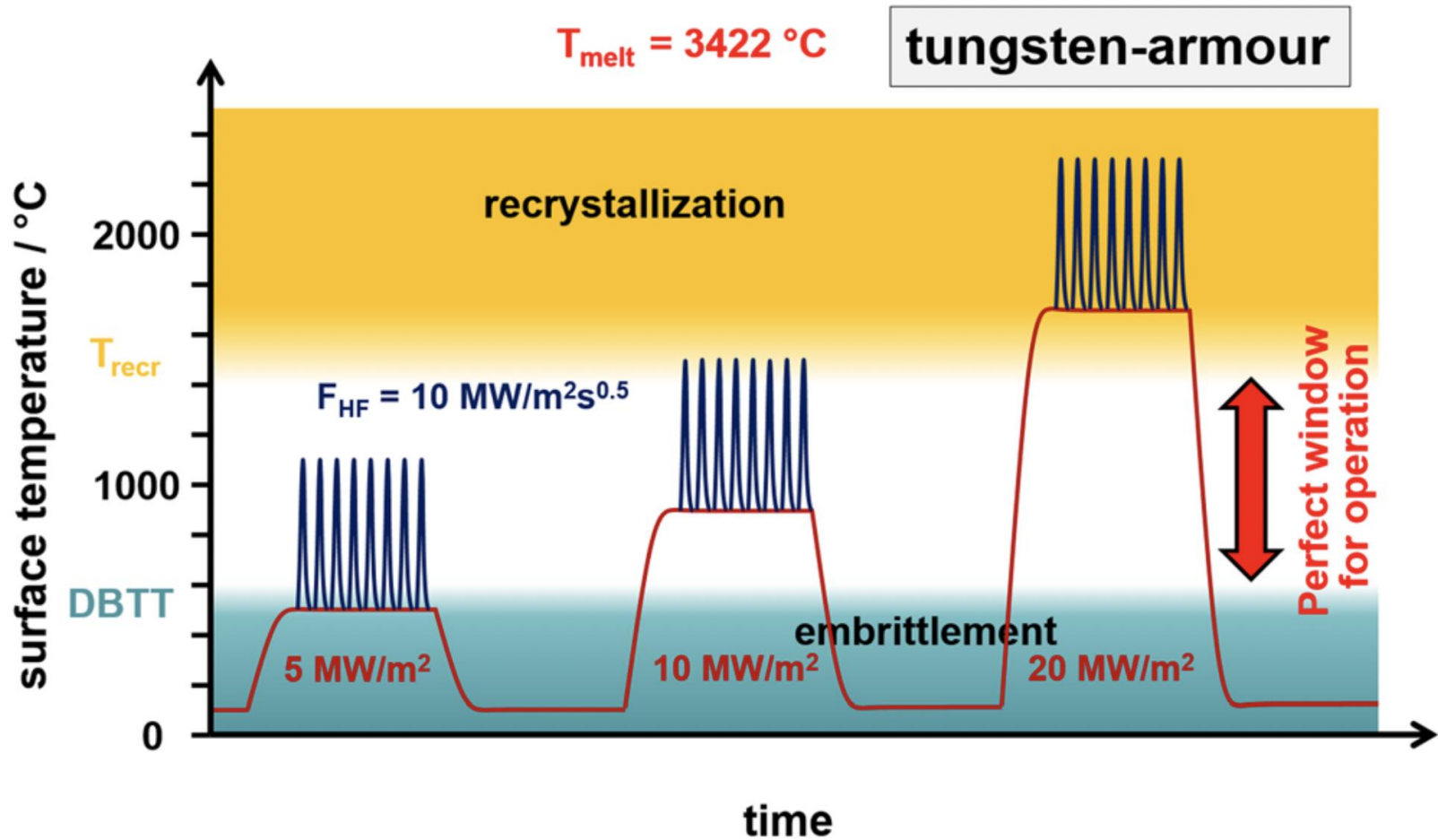
Sample failed under sequential loading at high T.



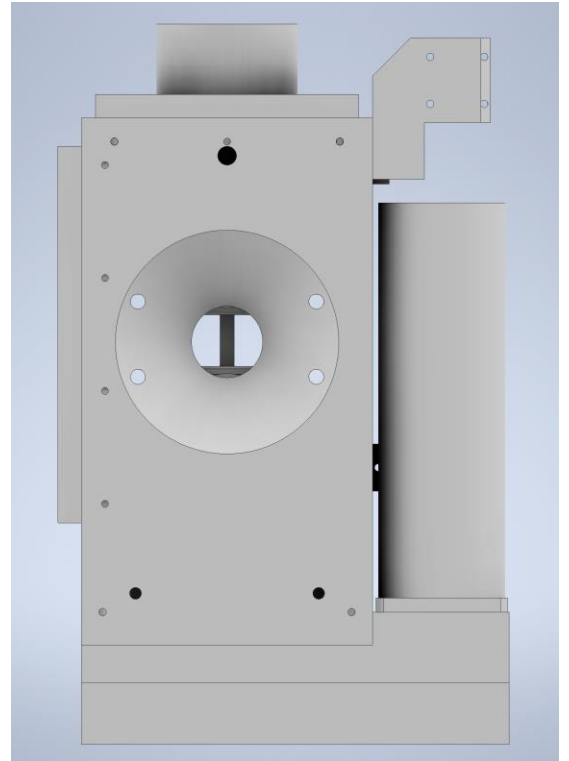
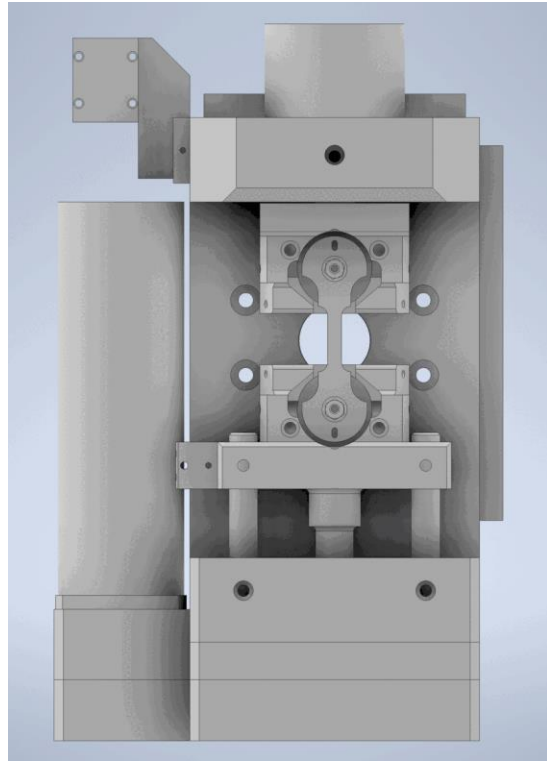
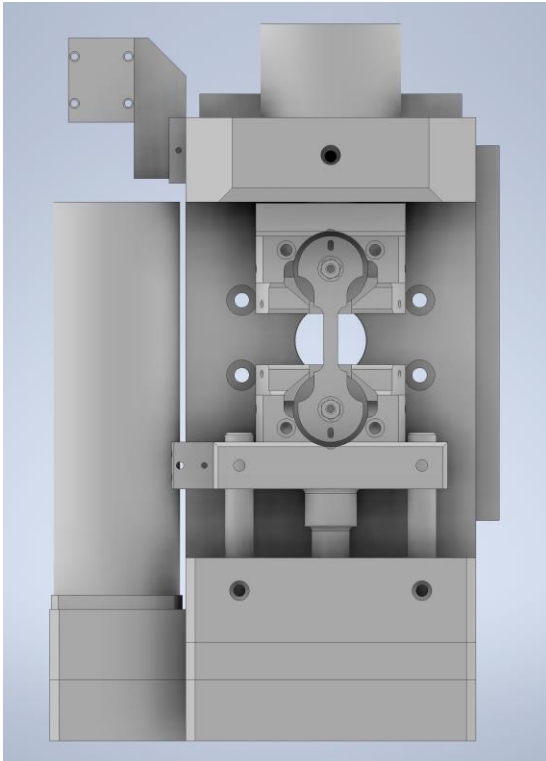
Paint speckle may affect emissivity of the sample.



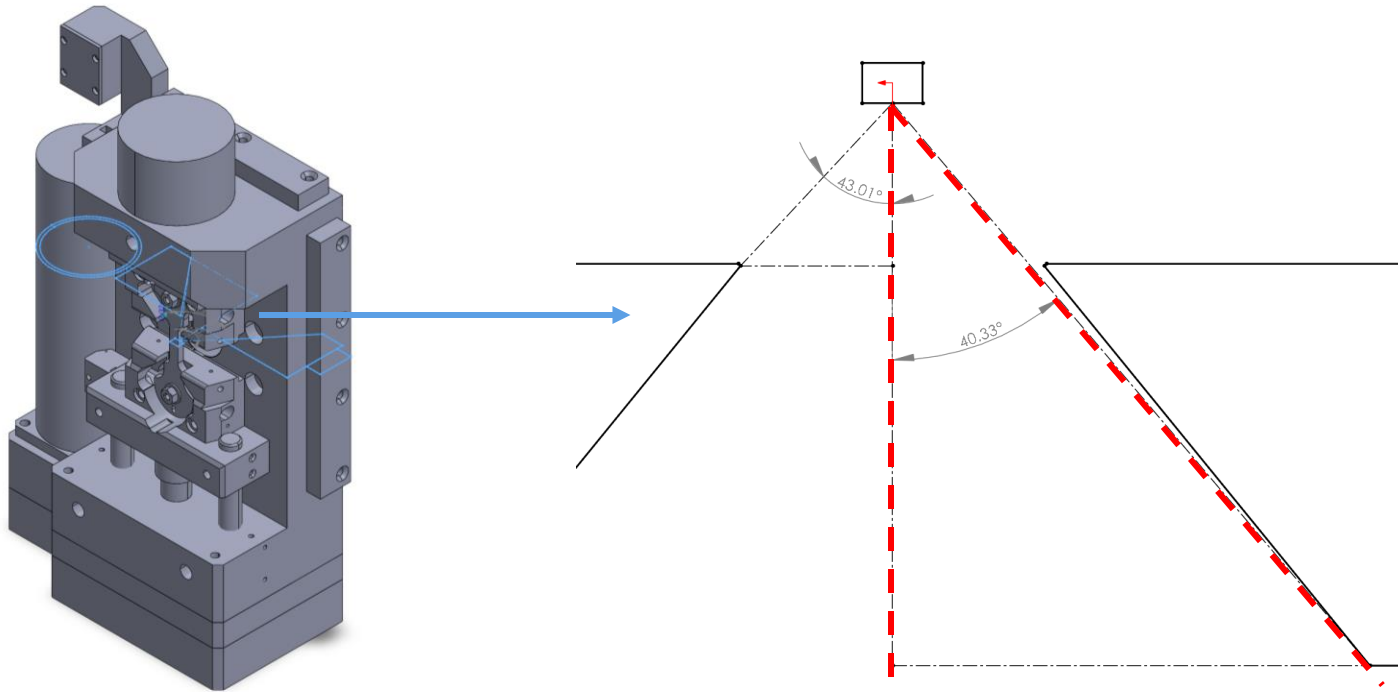
Fusion heat loads & thermal stresses will be cyclical.



NewTec MT1000 tensile rig



Constraints – FOV in Vertical Loading



Constraints – FOV in Horizontal Loading

