In Situ Ion Irradiated Creep & Mechanical Testing at the Michigan Ion Beam Laboratory

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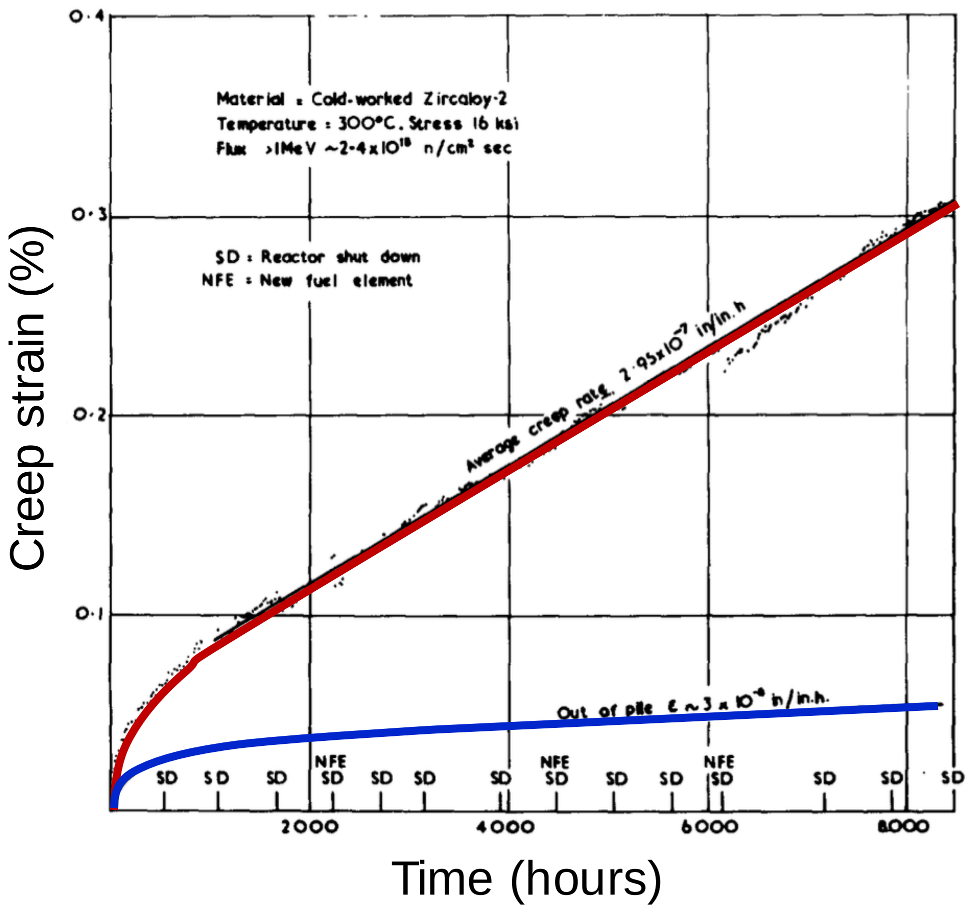
Nuclear Science User Facilities (NSUF) Annual Program Review
NSUF In Situ/Post-Irradiation Mechanical Testing/Corrosion Capabilities

Irradiation can increase creep rates significantly.



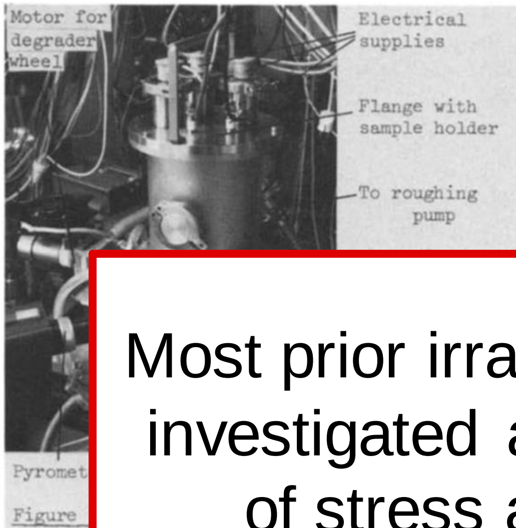
Irradiation creep
$\dot{\varepsilon} = 8.2 \times 10^{-11} s^{-1}$

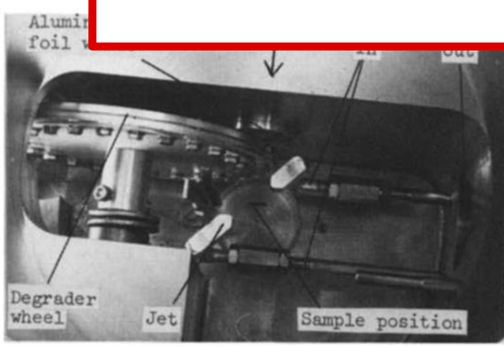
One order
of magnitude!

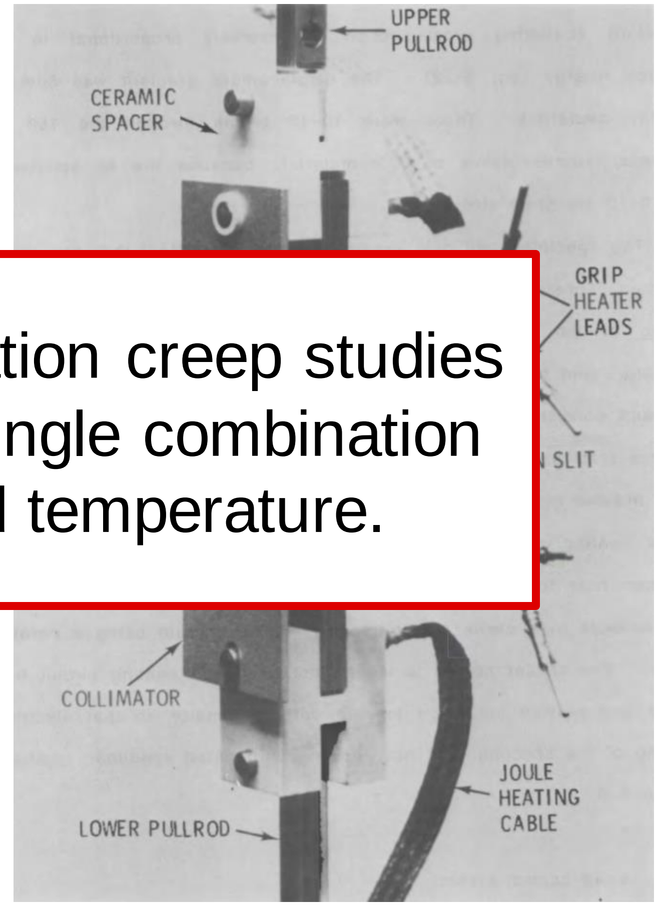
Thermal creep
$\dot{\varepsilon} = 8.3 \times 10^{-12} s^{-1}$

Harries, J. Nucl. Mater 65 (1977) 157-173

Ion-irradiation creep facilities can accelerate testing.





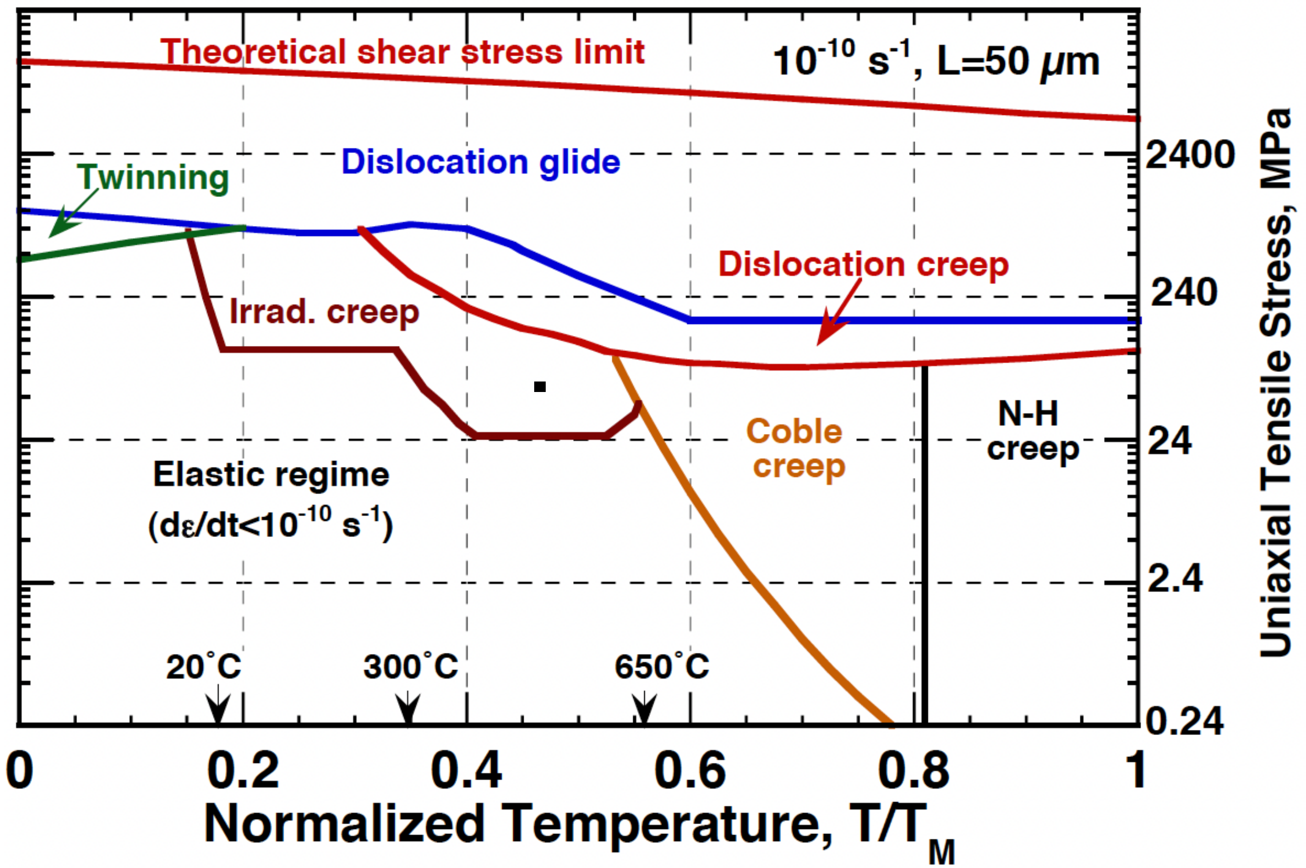


Most prior irradiation creep studies investigated a single combination of stress and temperature.

Schroeder and Batfalsky, J. Nucl. Mater. 103 (1981) 839
Henager, PhD Thesis, Univ. Washington (1984) PNL-5034 UC-25

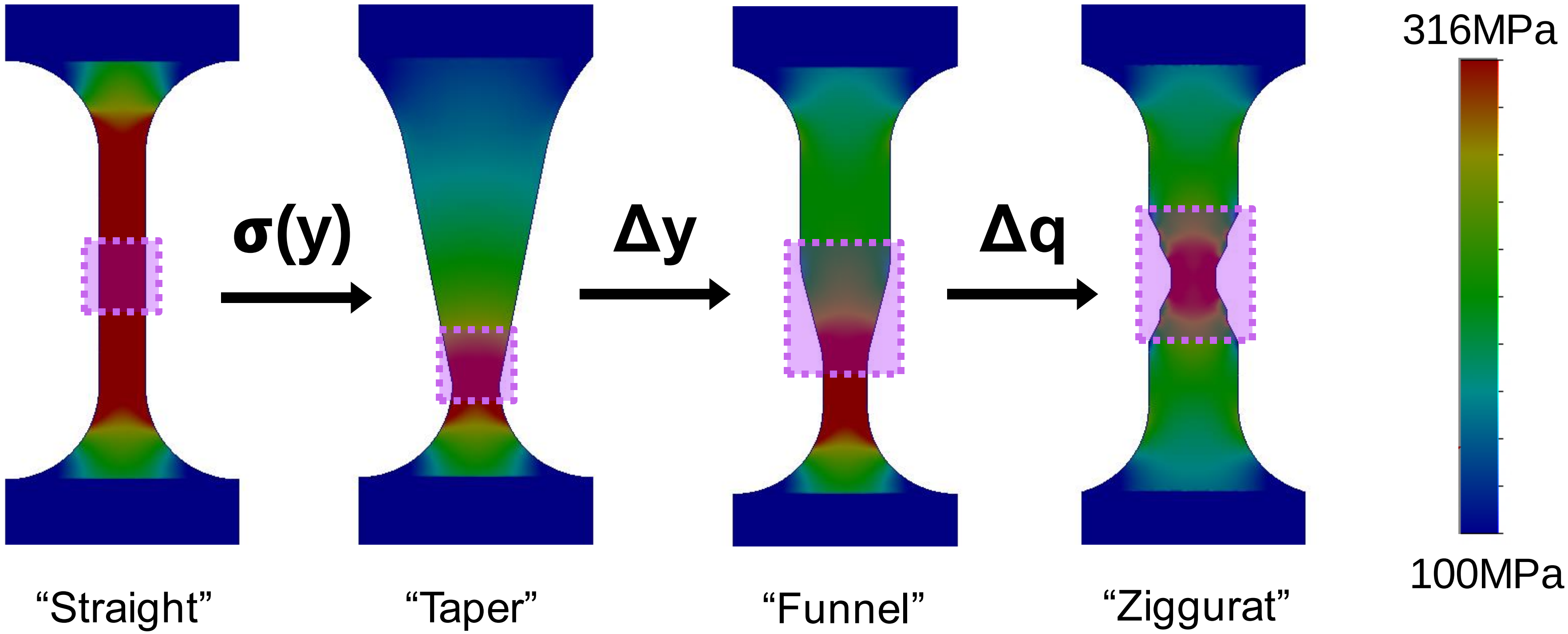
0D experiments are slow to explore parameter space.

Creep deformation mechanism map for 316 stainless steel.



Zinkle and Lucas, Fusion Mater. Semiann. Progress Report (2003) DOE/ER-0313/34

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



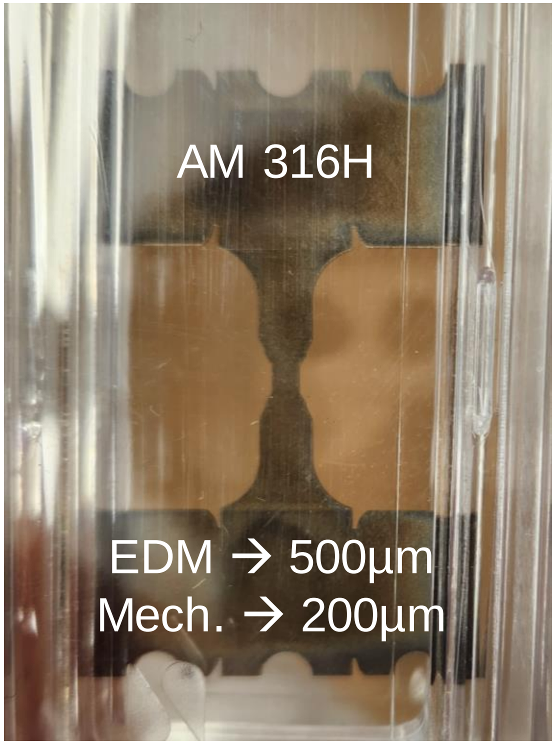
Material: 25 µm-thick annealed 316 SS foil

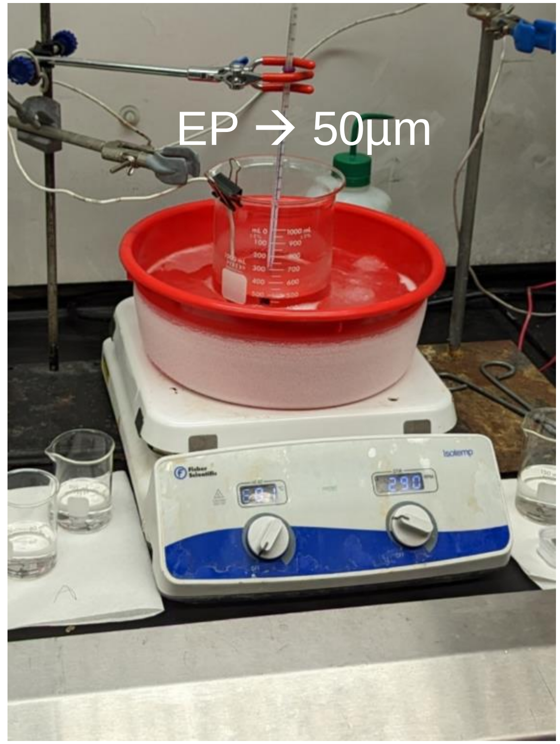
Stress: 158-316 MPa, **Temperature:** 550°C

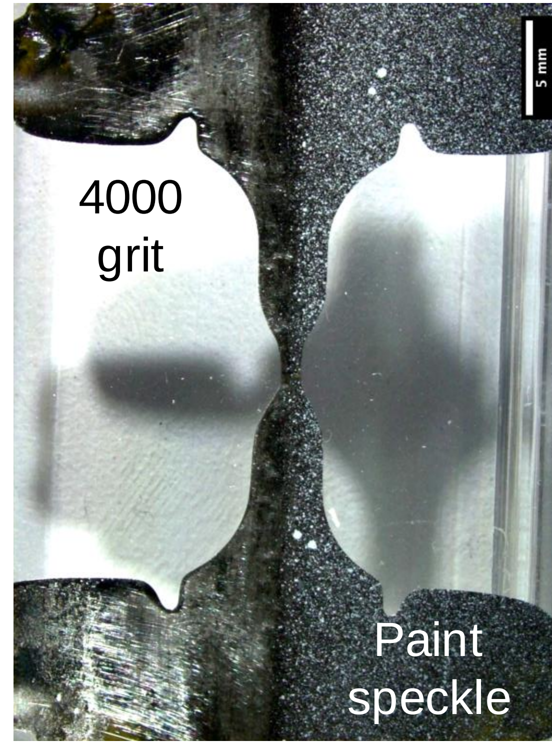
3 MeV H+ irradiation area

Credit: Wyatt Peterson

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



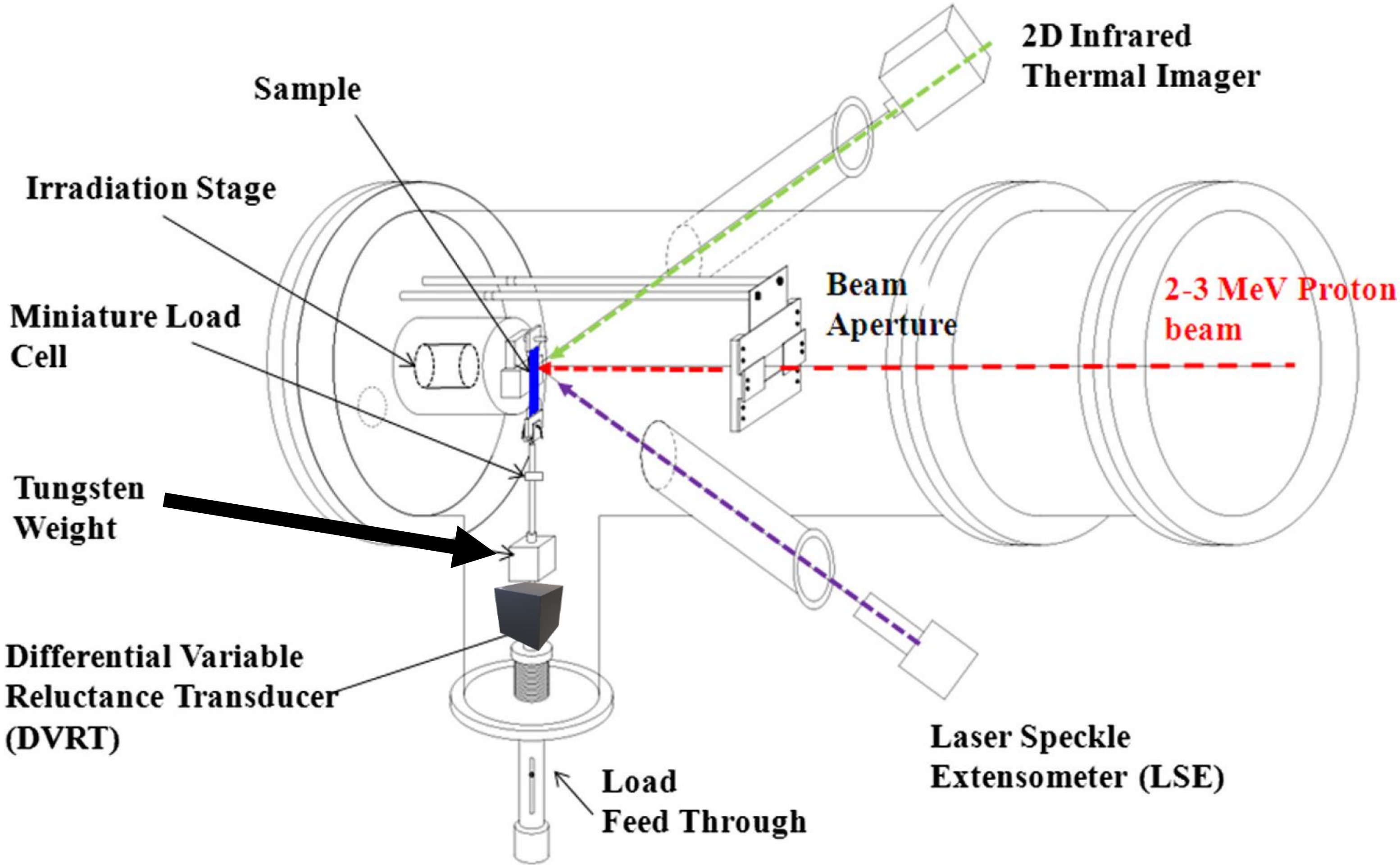




RTE #4654 – 'Quantifying the effect of simultaneous vs. sequential irradiation on creep performance of additively manufactured austenitic stainless steel' (PI Massey).

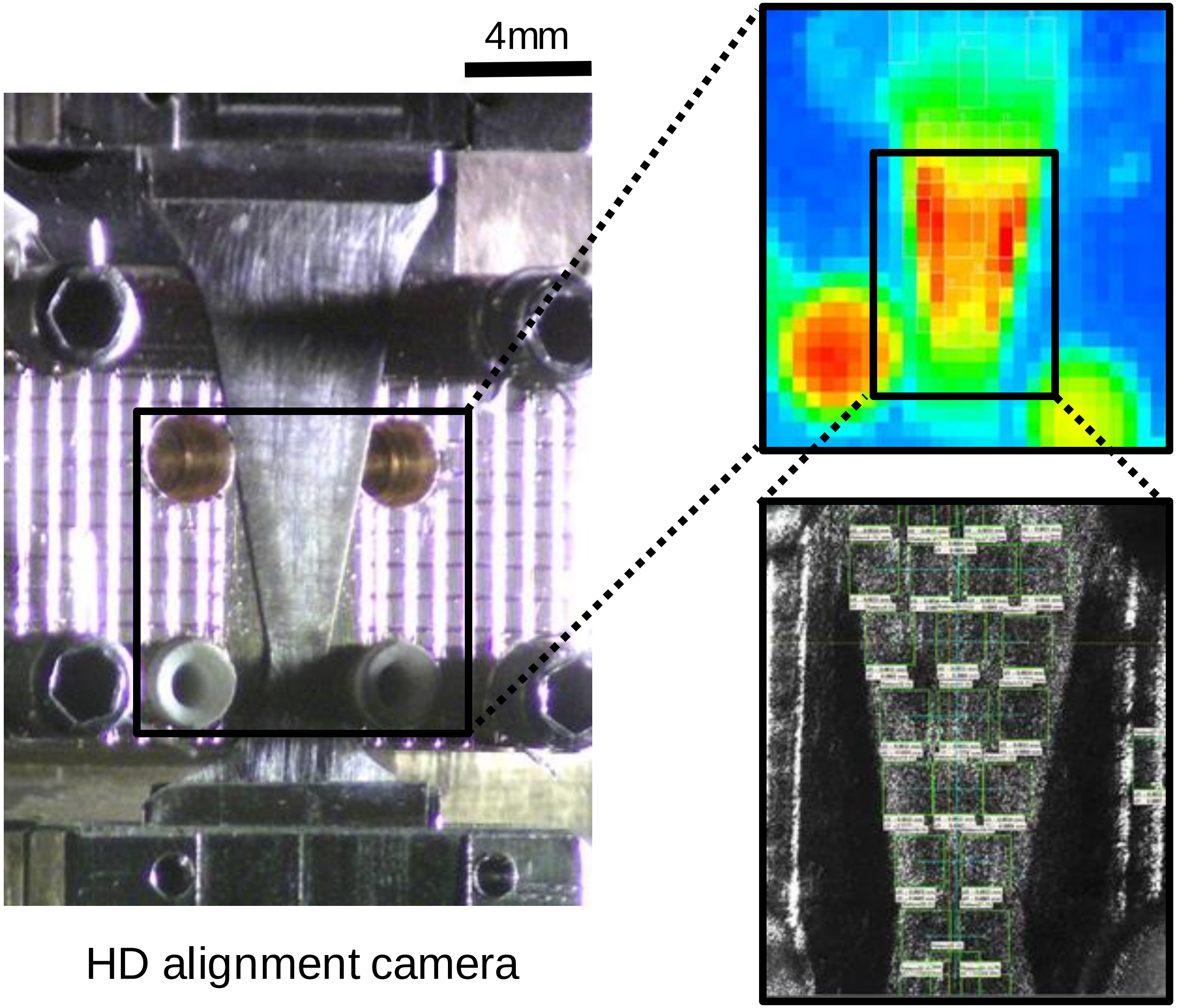
RTE #4817 – 'Investigating the evolution of M23C6 and MX-type precipitates in additively manufactured Grade 91 steel under high T. simultaneous & sequential stress & irradiation' (PI Narra)

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



Xu et al. J. Nucl. Mater. 441 (2013) 681-687

Imaging captures local temperature & strain fields.



HD alignment camera

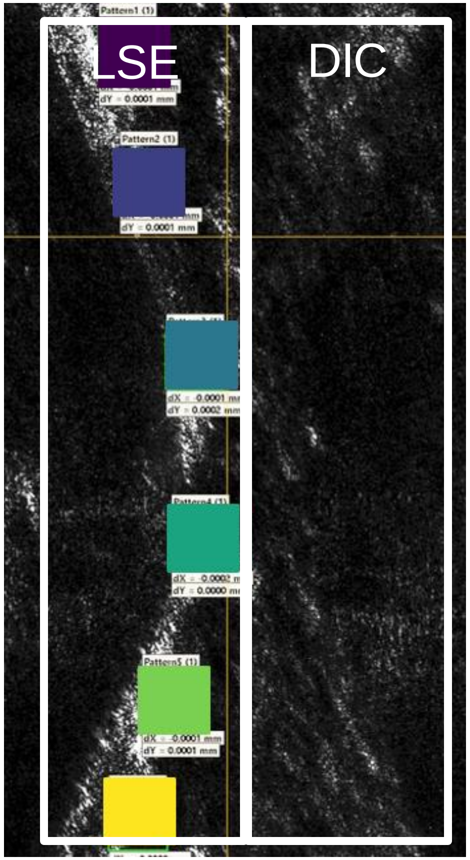
FLIR infrared camera

T(x,y)

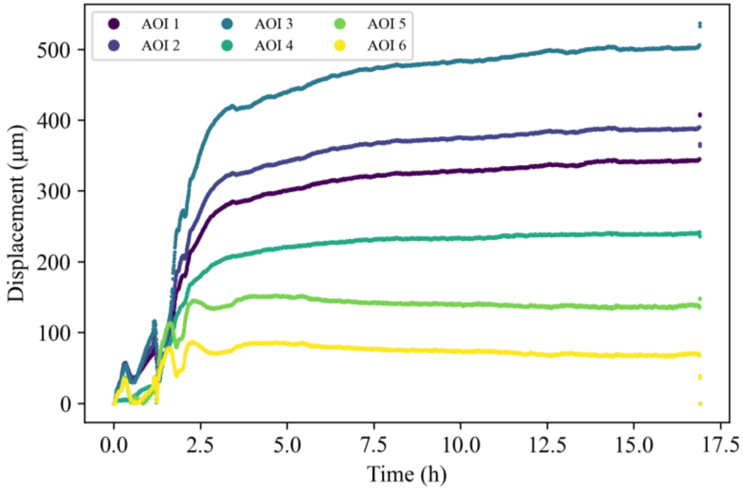
Zwick Roell LSE

ε(x,y)

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

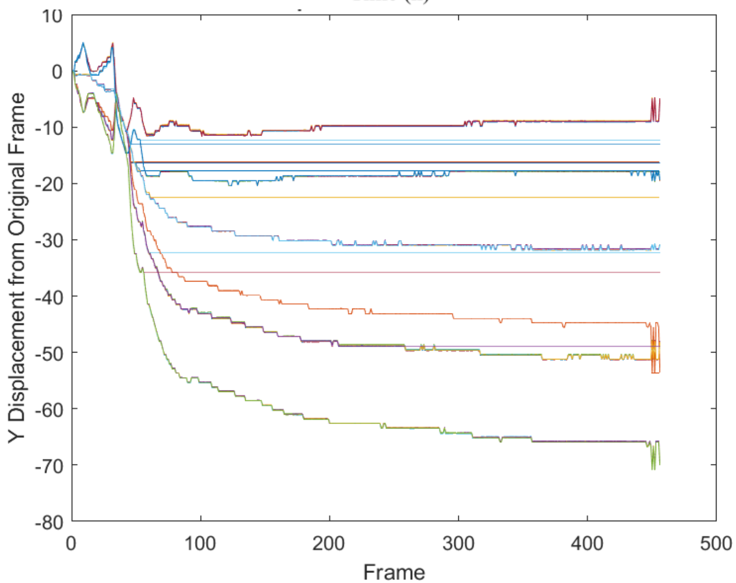


“Ziggurat” sample



Laser Speckle Extensometer (LSE) Displacement

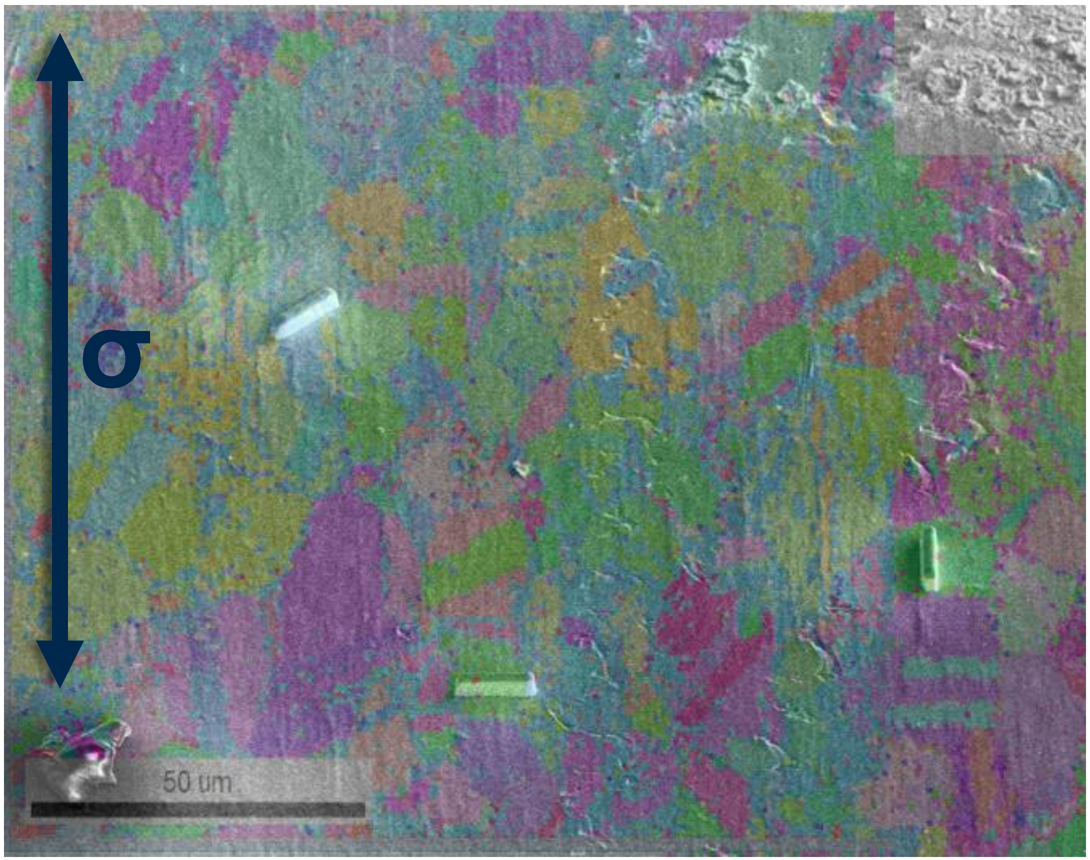
≈

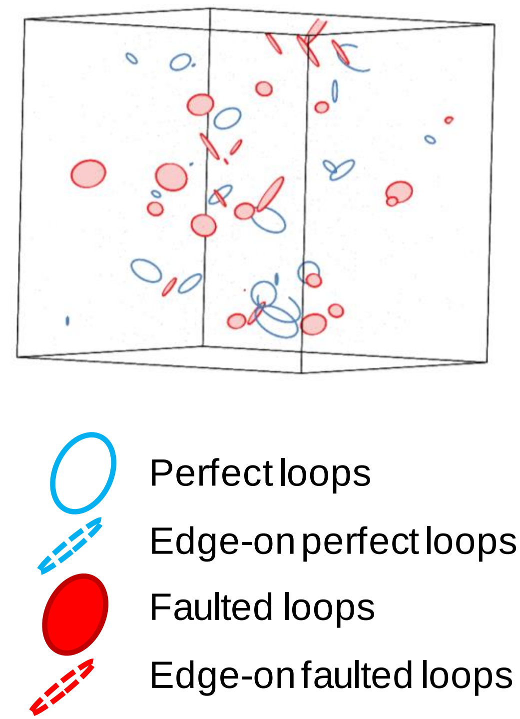


Digital Image Correlation (DIC) Displacement

Credit: Mackenzie Warwick & Ben Arms

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



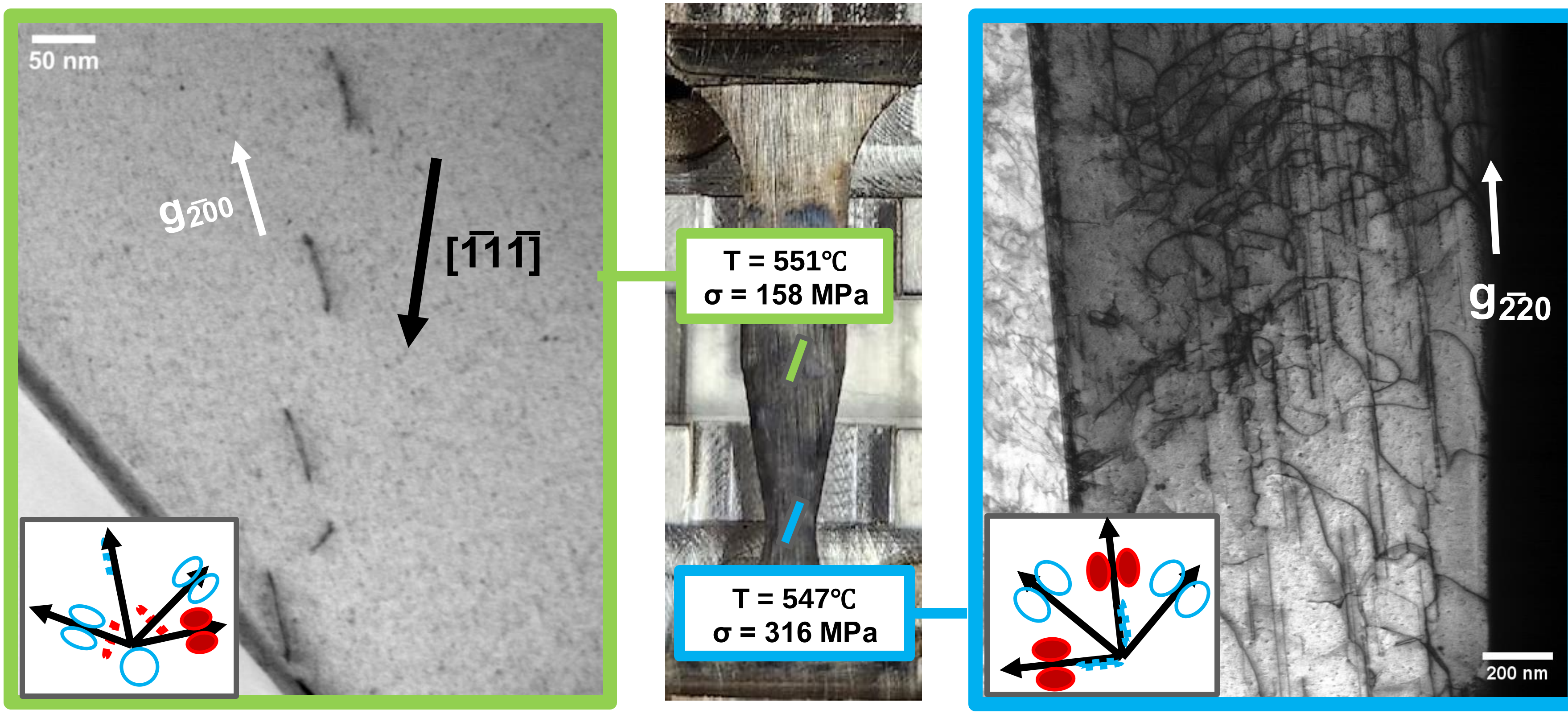


Credit: Mackenzie Warwick

Initial characterization shows distinct microstructures.

158 MPa

316 MPa



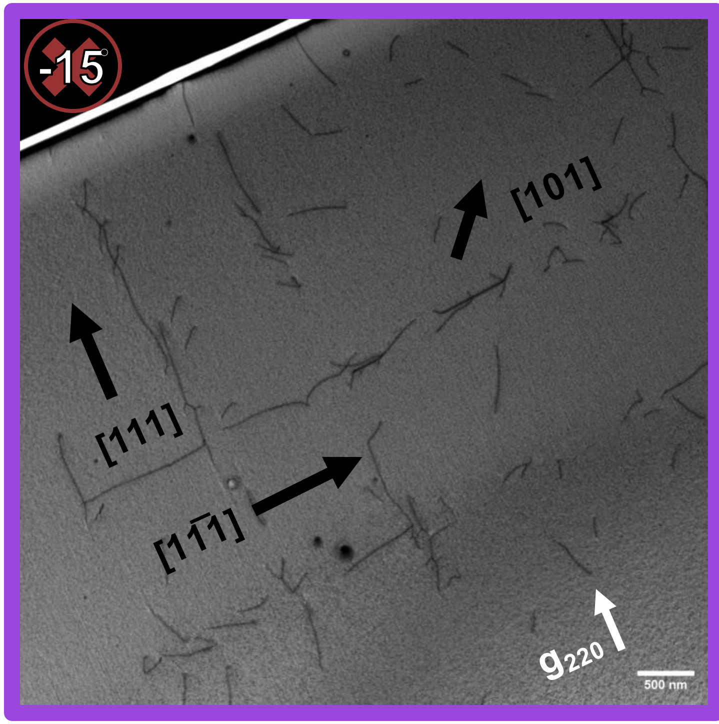
On-Zone S/TEM (011)
Average length: 59.3 ± 7.1 nm

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

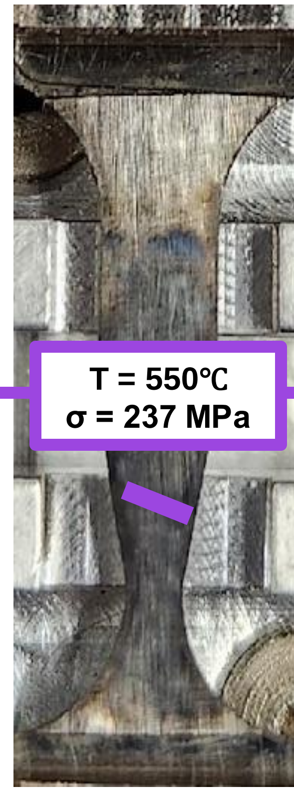
Credit: Mackenzie Warwick

Further TEM characterization is in progress...

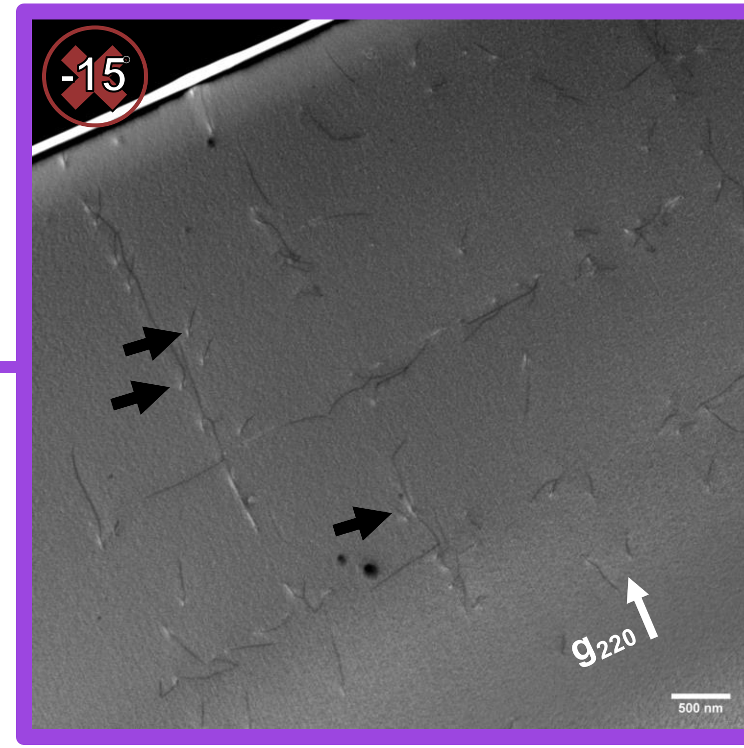
237 MPa



On-zone S/TEM BF <001>



237 MPa

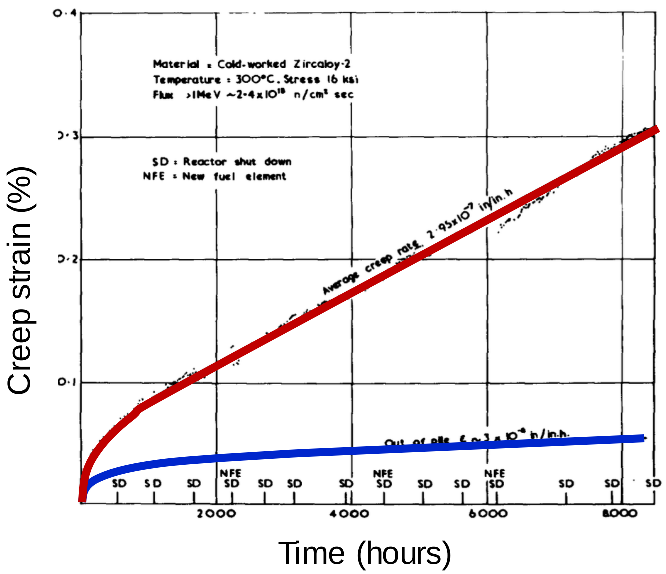


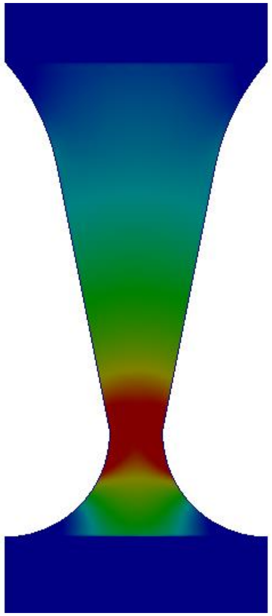
On-zone S/TEM DF <001>

Now characterization has become the bottleneck!

Credit: Mackenzie Warwick

In Situ Ion Irradiated Creep & Mechanical Testing at the Michigan Ion Beam Laboratory



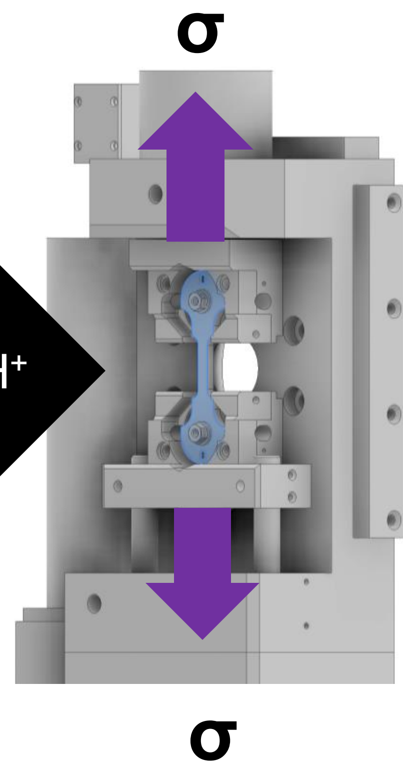


Simultaneous ion-irradiation creep fatigue (ICF) will be investigated at the Michigan Ion Beam Lab*

NewTec MT1000
in-situ tensile stage

Grip heating up to 1000ºC

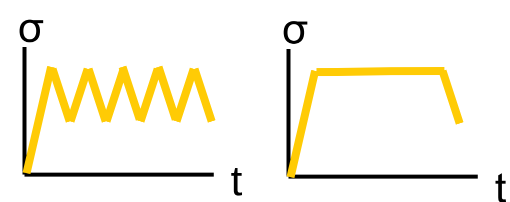
IRRADIATION 3 MeV H^+



T > $0.4T_m$ = CREEP

σ(t) = FATIGUE

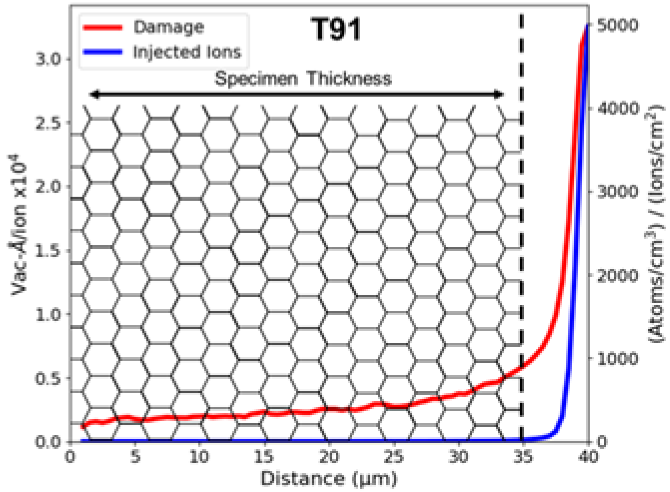
in situ and
in operando
tensile testing

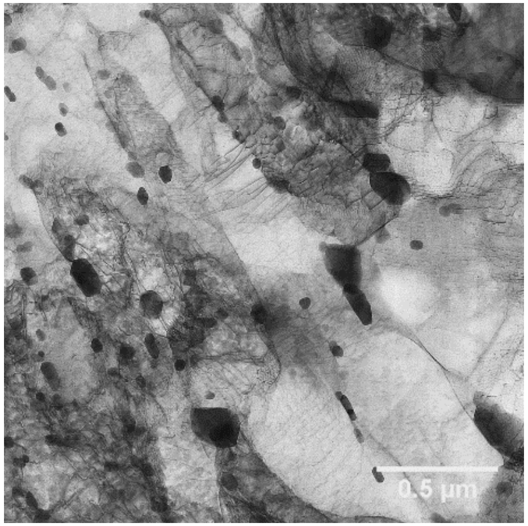


CAD courtesy of NewTec Scientific

* and at the Wisconsin Ion Beam Lab!

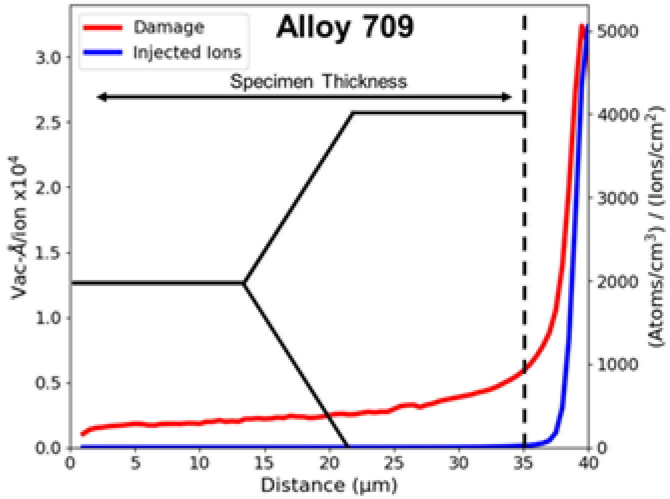
Specimen thickness & microstructural length scale are critical parameters.

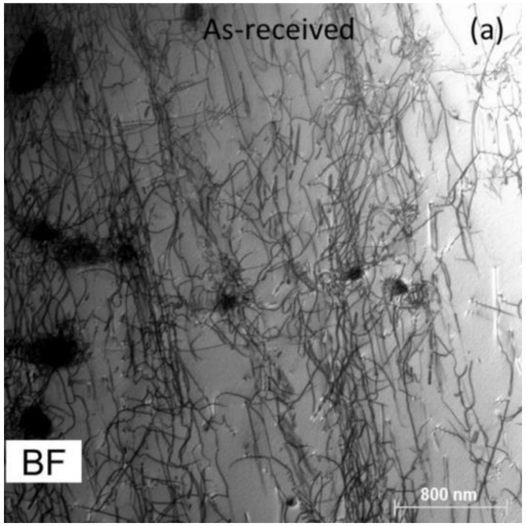




Grade 91

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

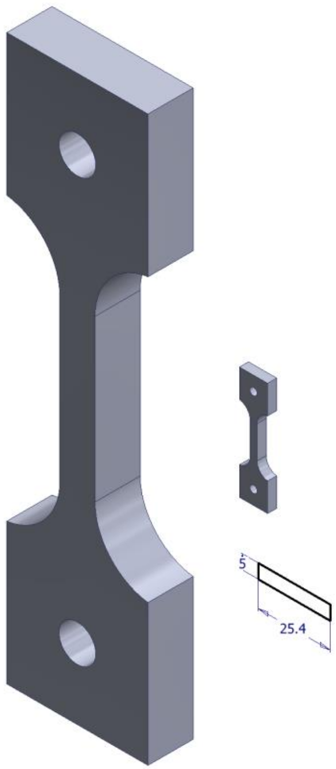


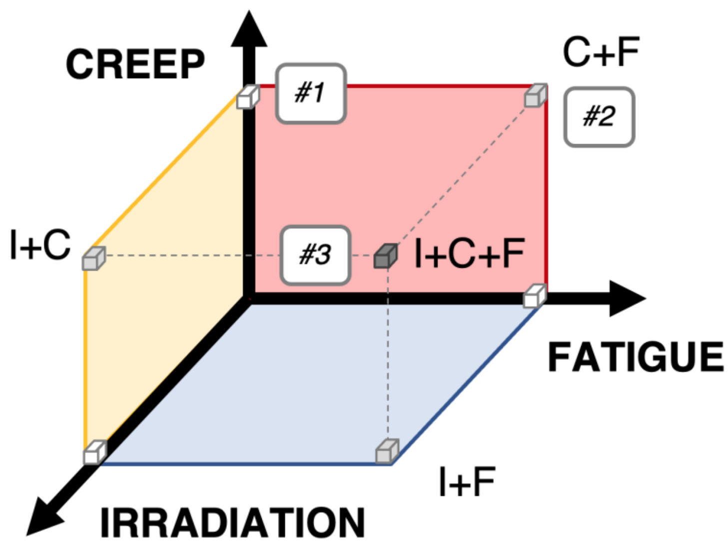


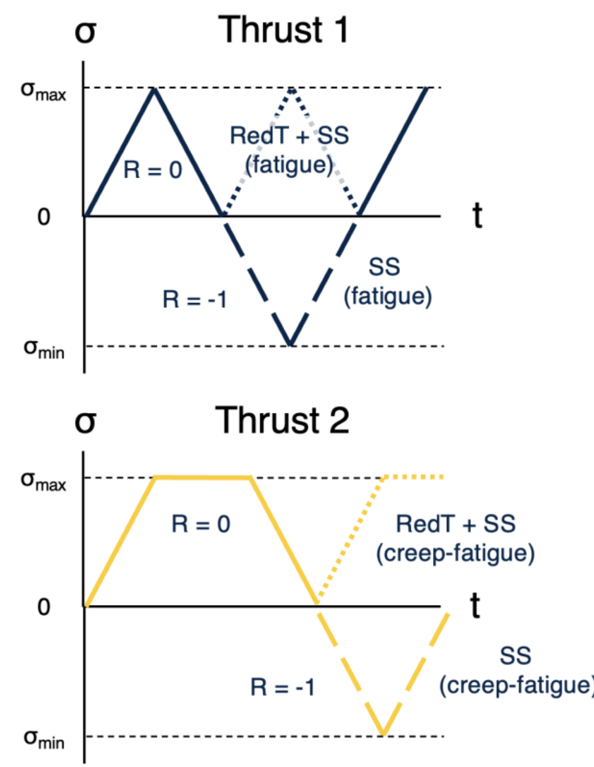
Alloy 709

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

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



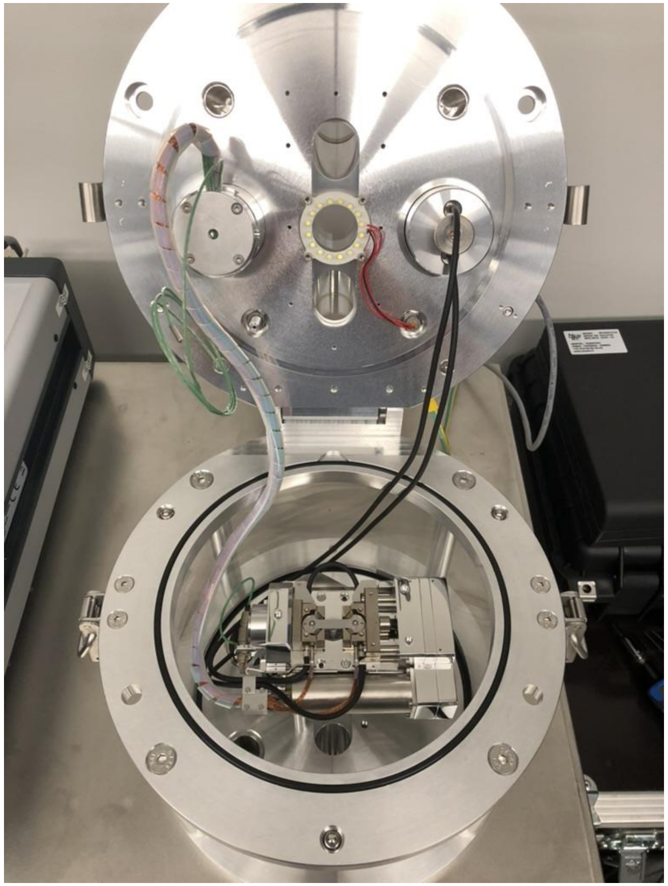




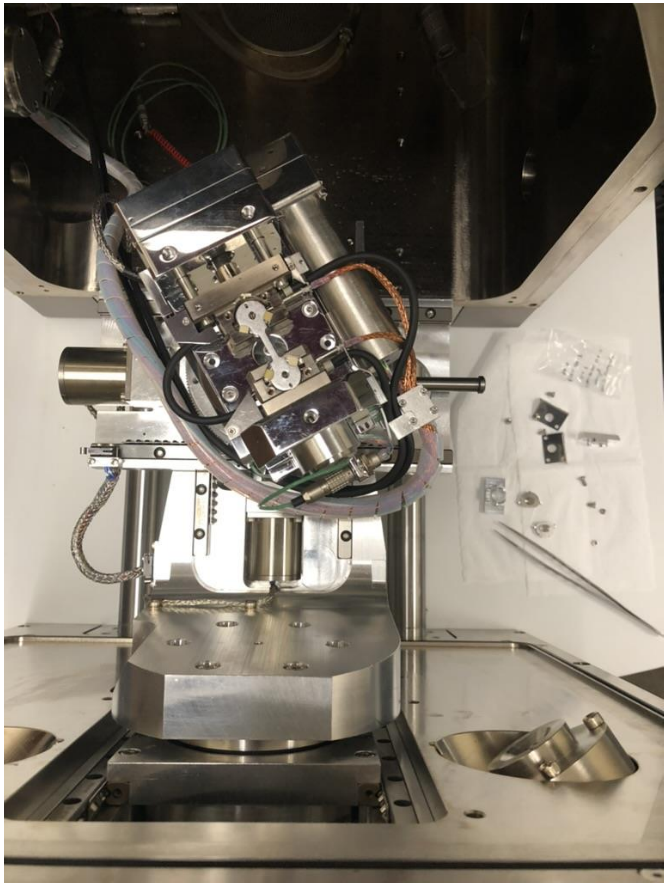
Standard size (SS)
Reduced-thickness (RedT)

Stress intensity ratio (R)
Hold time under load.

Miniature tensile rig received and tested within SEM.

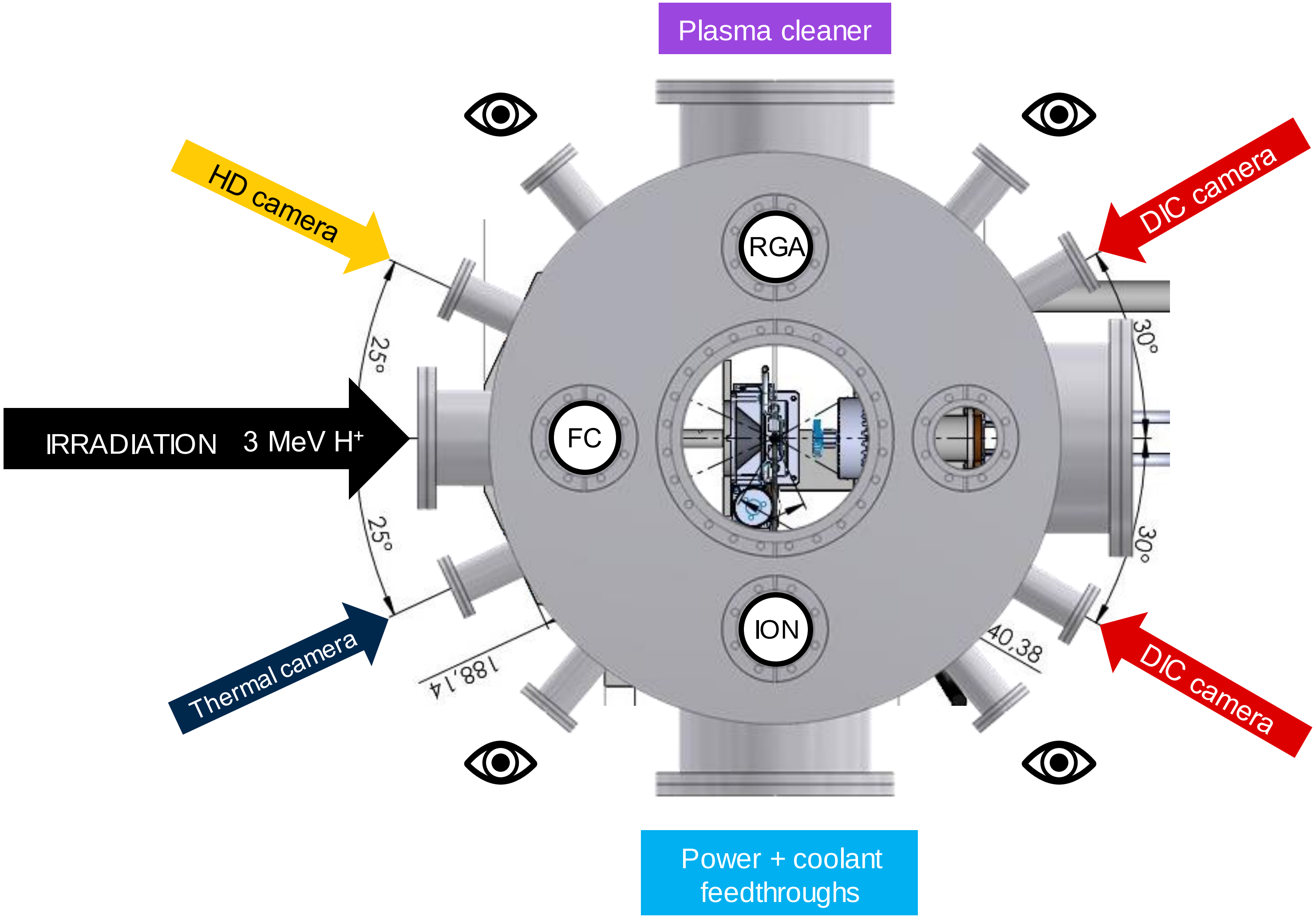


MT1000 within exoSEM



within TESCAN MIRA3

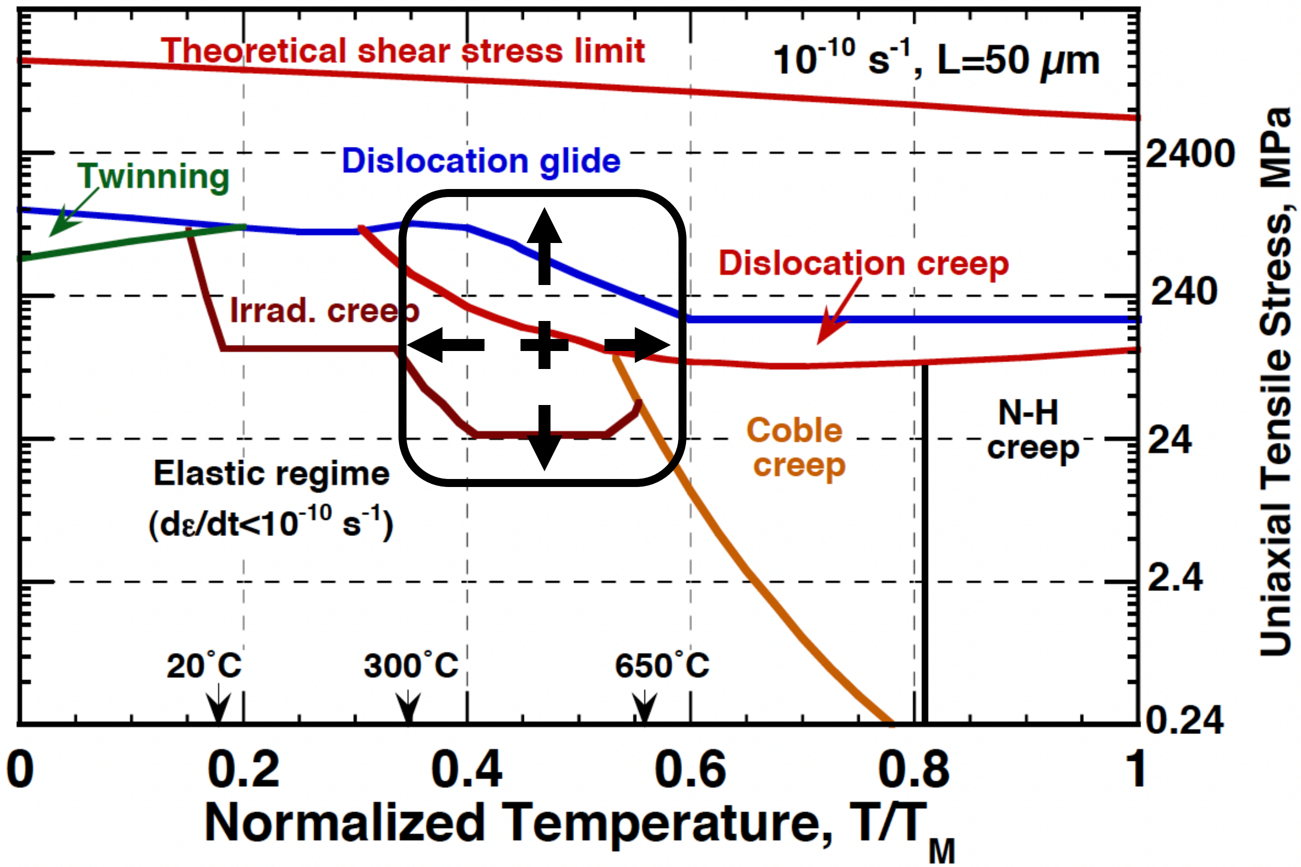
Beamline chamber in the process of being designed.



Drawing courtesy of NewTec Scientific

1D gradient experiments will accelerate exploration.

Creep deformation mechanism map for 316 stainless steel.



Zinkle and Lucas, Fusion Mater. Semiann. Progress Report (2003) DOE/ER-0313/34

Acknowledgements

RTE-23-4654
Quantifying the effect of simultaneous vs. sequential irradiation on creep performance of additively manufactured austenitic stainless steel.

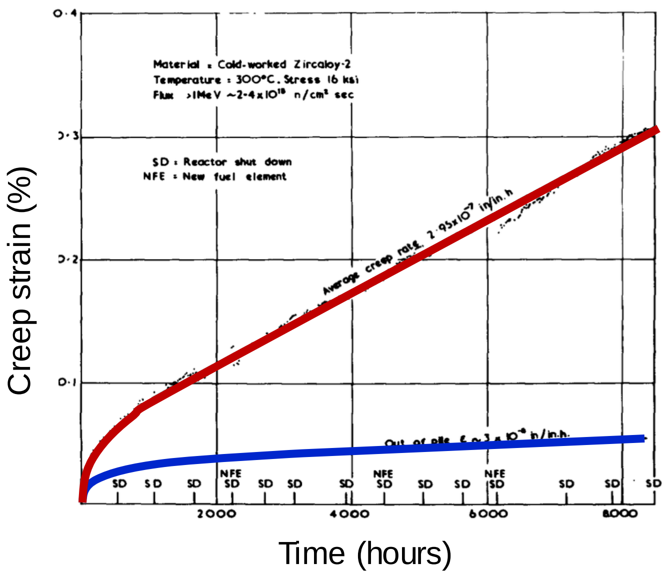
RTE-23-4654
Investigating the evolution of M23C6 and MX-type precipitates in additively manufactured Grade 91 steel under high T. simultaneous & sequential stress & irradiation.

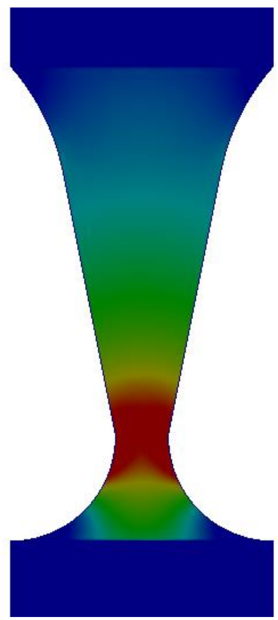
CFA-22-27043
Accelerated irradiation creep testing coupled with self-adaptive accelerated molecular dynamics simulations for scalability analysis.

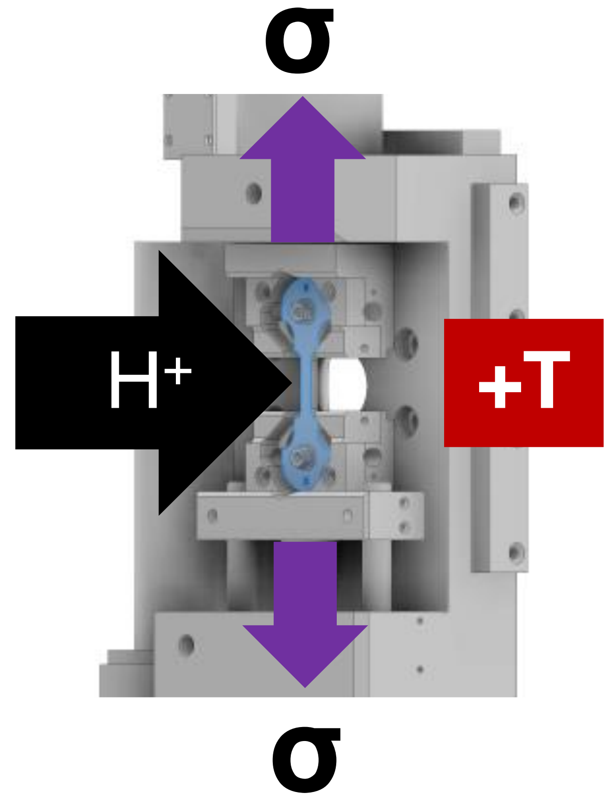
CFA-23-29058
Mechanism driven evaluations of sequential and simultaneous irradiation-creep-fatigue testing.

GSI-24-32151
In situ ion irradiation testing facilities for the investigation of nuclear materials under mechanical and thermal extremes

In Situ Ion Irradiated Creep & Mechanical Testing at the Michigan Ion Beam Laboratory

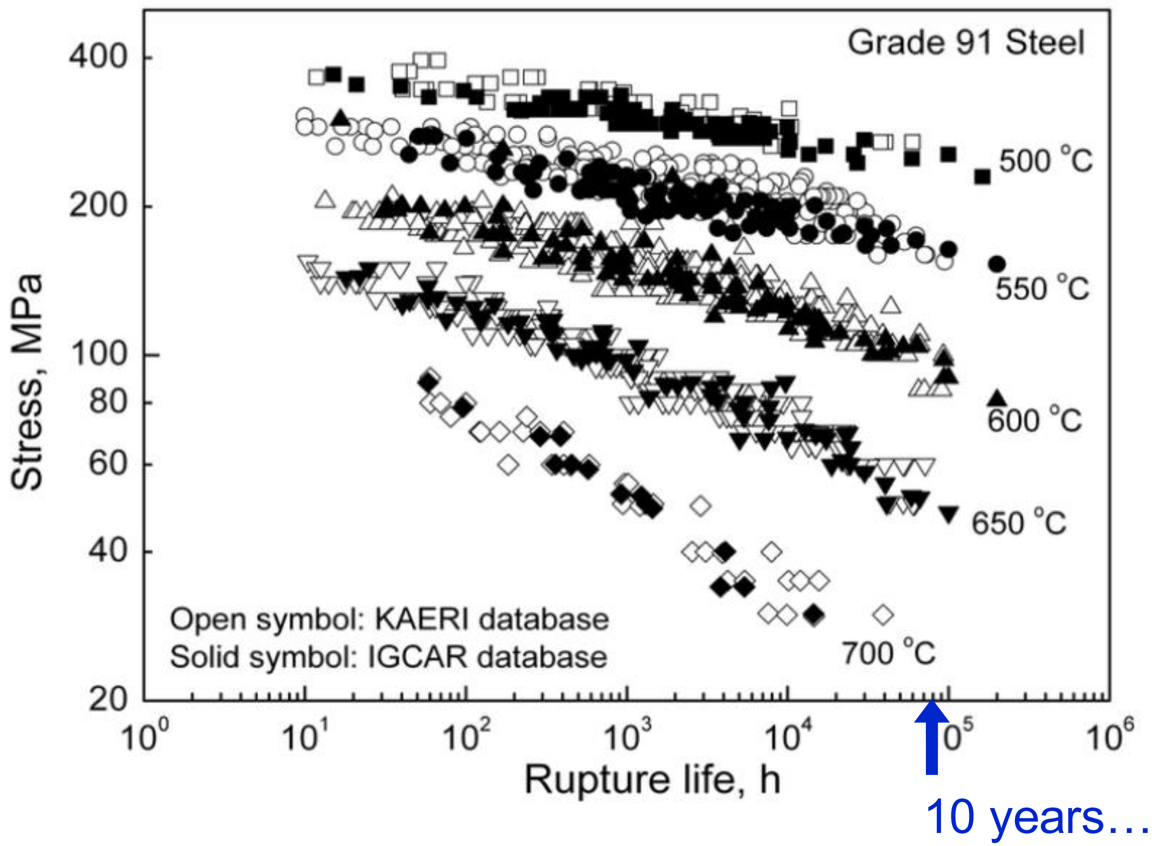






Nuclear Science User Facilities (NSUF) Annual Program Review
NSUF In Situ/Post-Irradiation Mechanical Testing/Corrosion Capabilities

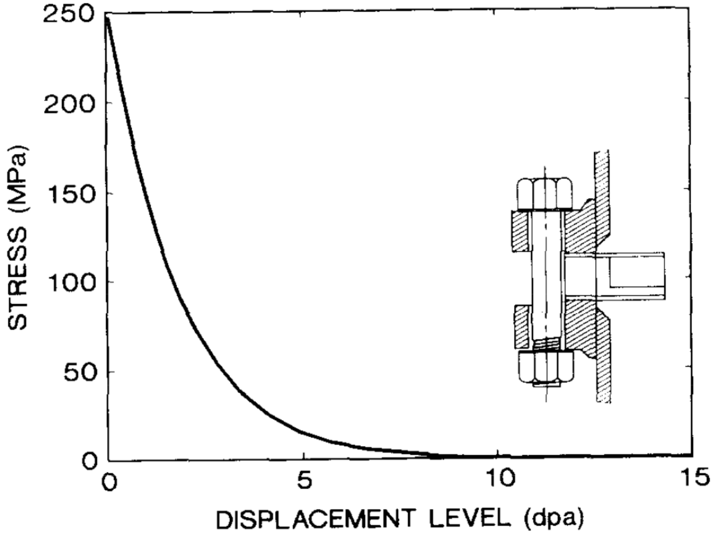
Elevated temperatures & stress cause thermal creep.

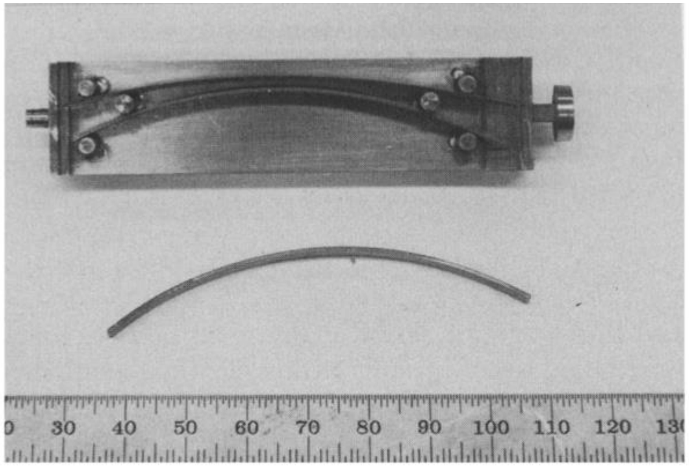


Increasing temperatures decrease the lifetime.

Choudary et al. Procedia Eng. 86 (2014) 335 – 341

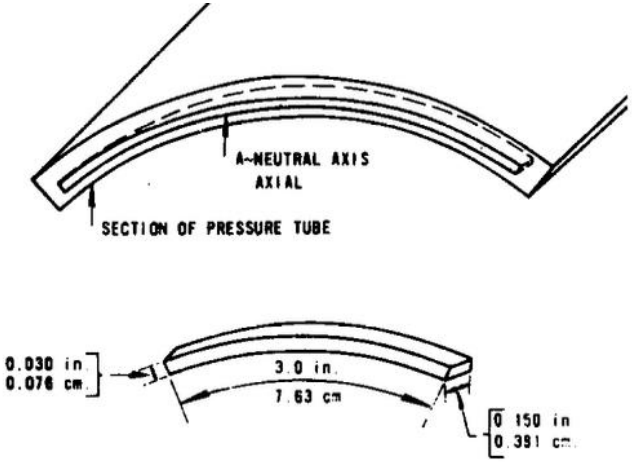
Stress relaxation measurements are inaccurate at long times and difficult to measure in-reactor.





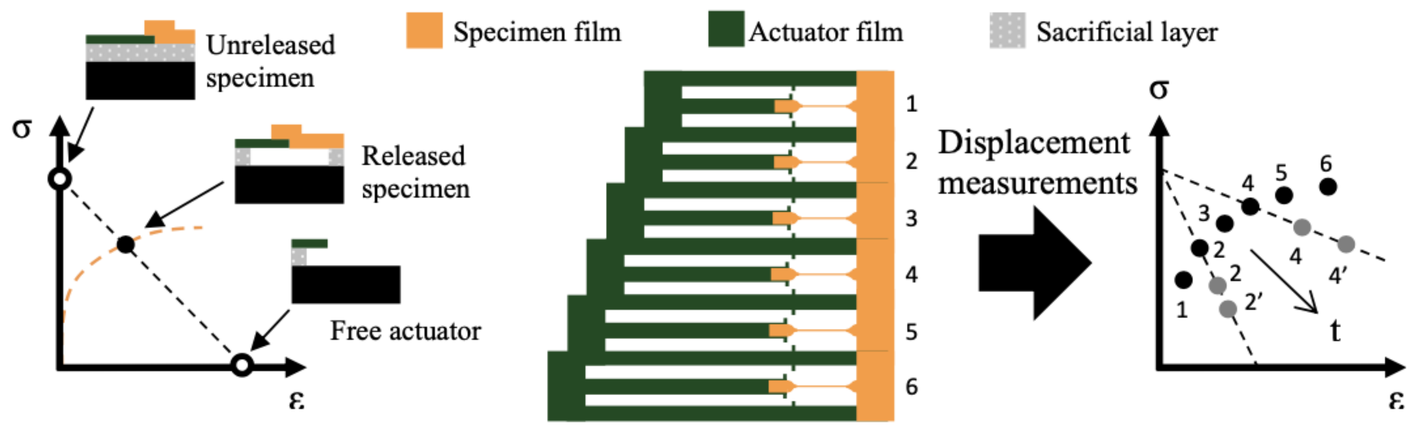
$$\dot{\varepsilon} \propto \sigma(t)$$

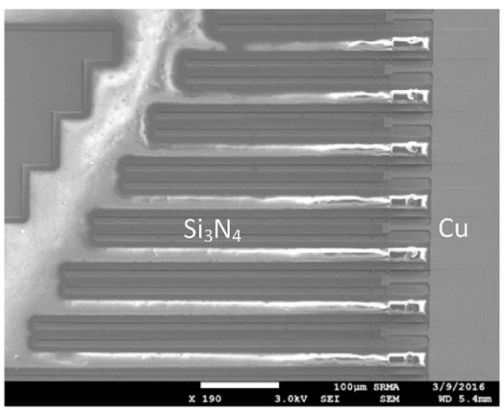
strain rate depends on stress which varies over time.

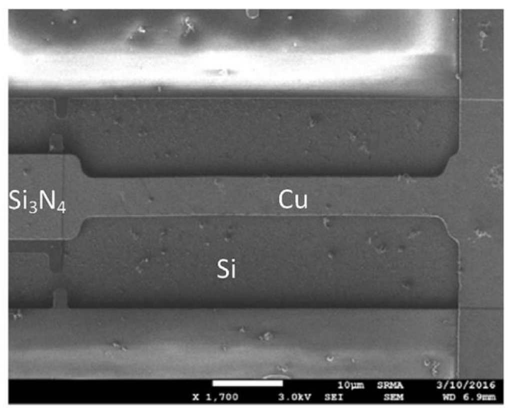


Grossbeck and Mansur, J. Nucl. Mater. 179 (1991) 130
Causey et al. J. Nucl. Mater. 159 (1988) 101

MEMS irradiation creep can perform parallel tests.



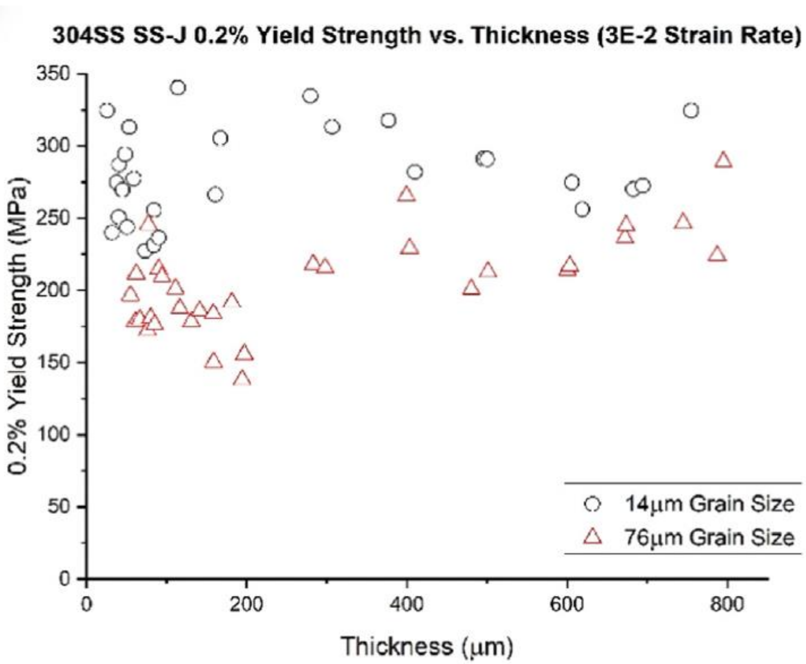
a)


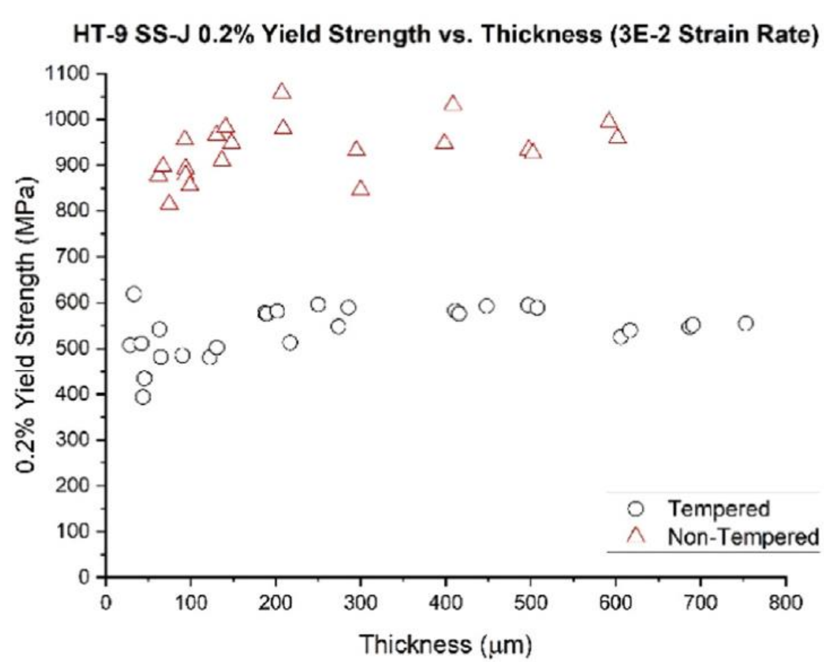
b)


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

Lapouge et al., J. Nucl. Mater. 476 (2016) 20

Yield stress can be obtained from 30μm samples.





Dong et al., J. Nucl. Mater. 575 (2023) 154207

Yield stress accurate for thickness/grain size > 1.

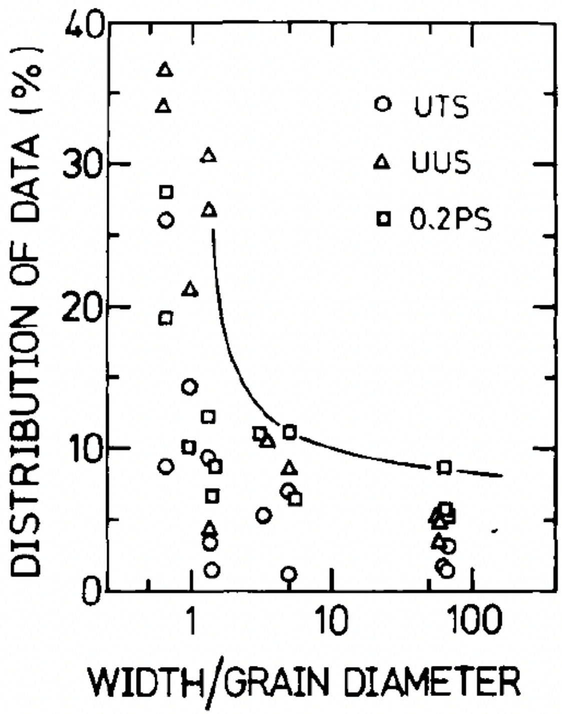


Fig. 1. The distribution of 0.2% proof stress (0.2PS), ultimate tensile strength (UTS), and ultimate uniform strain (UUS) as a function of w/d.

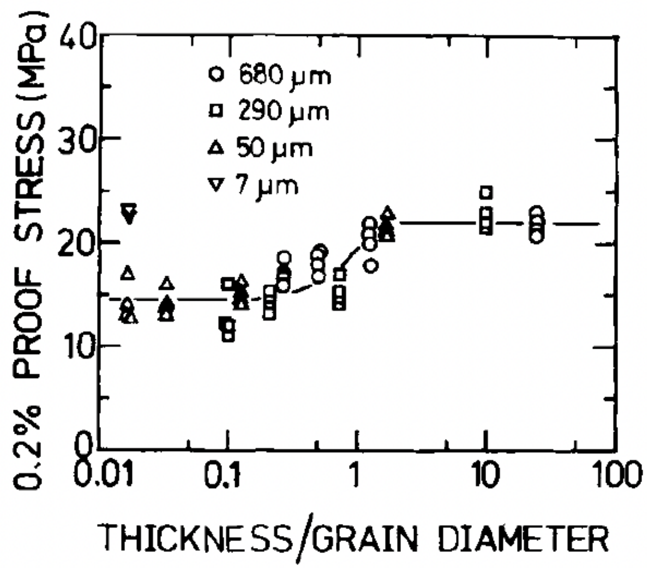


Fig. 2. The t/d dependence of 0.2% proof stress.

Igata et al., Proc. Intl. Conf. Strength Metals Alloys (1985) 39-43

Electropolishing – Methodology

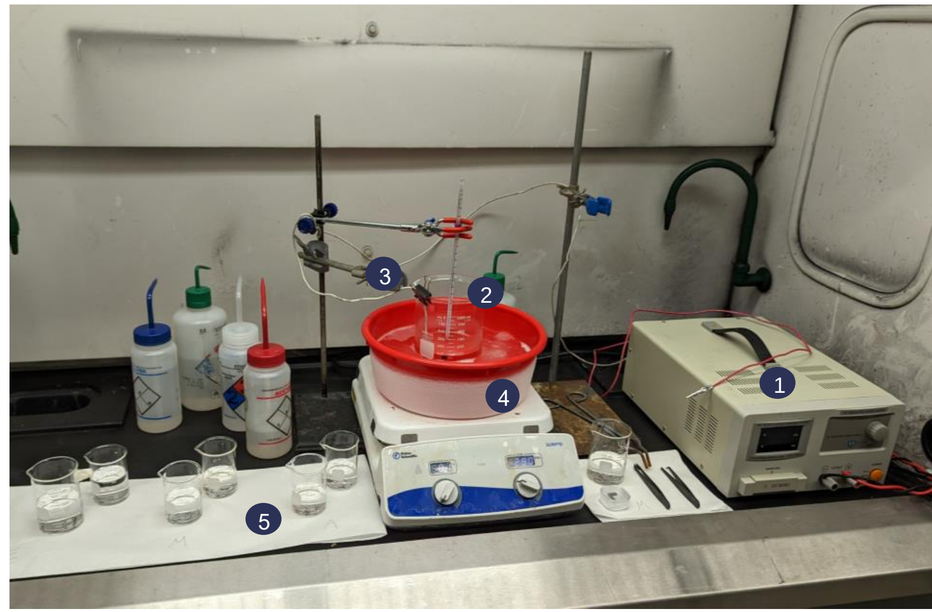
❶ Voltage source, set to 40V

❷ Acid solution; 10% Perchloric acid, 90% Methanol

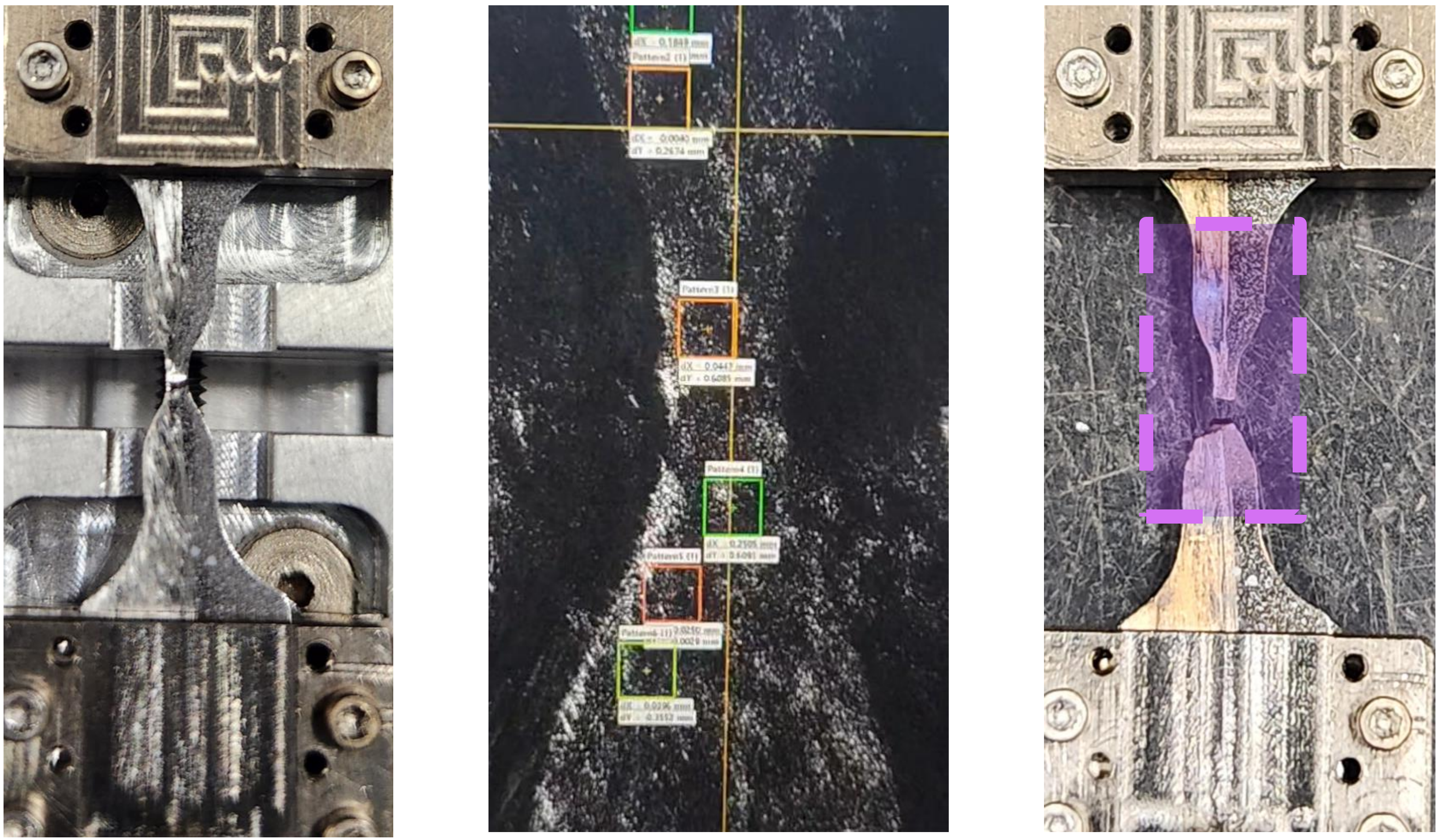
❸ Negative lead, connected to platinum mesh

❹ Methanol bath, cool to -45 °C

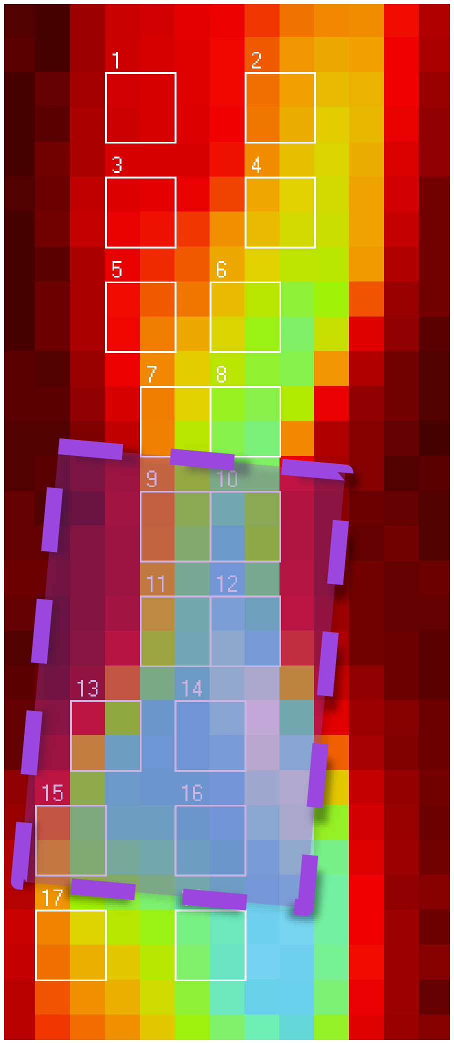
❺ Sample cleaning process; Acetone, Methanol, and Ethanol for 20 seconds twice each

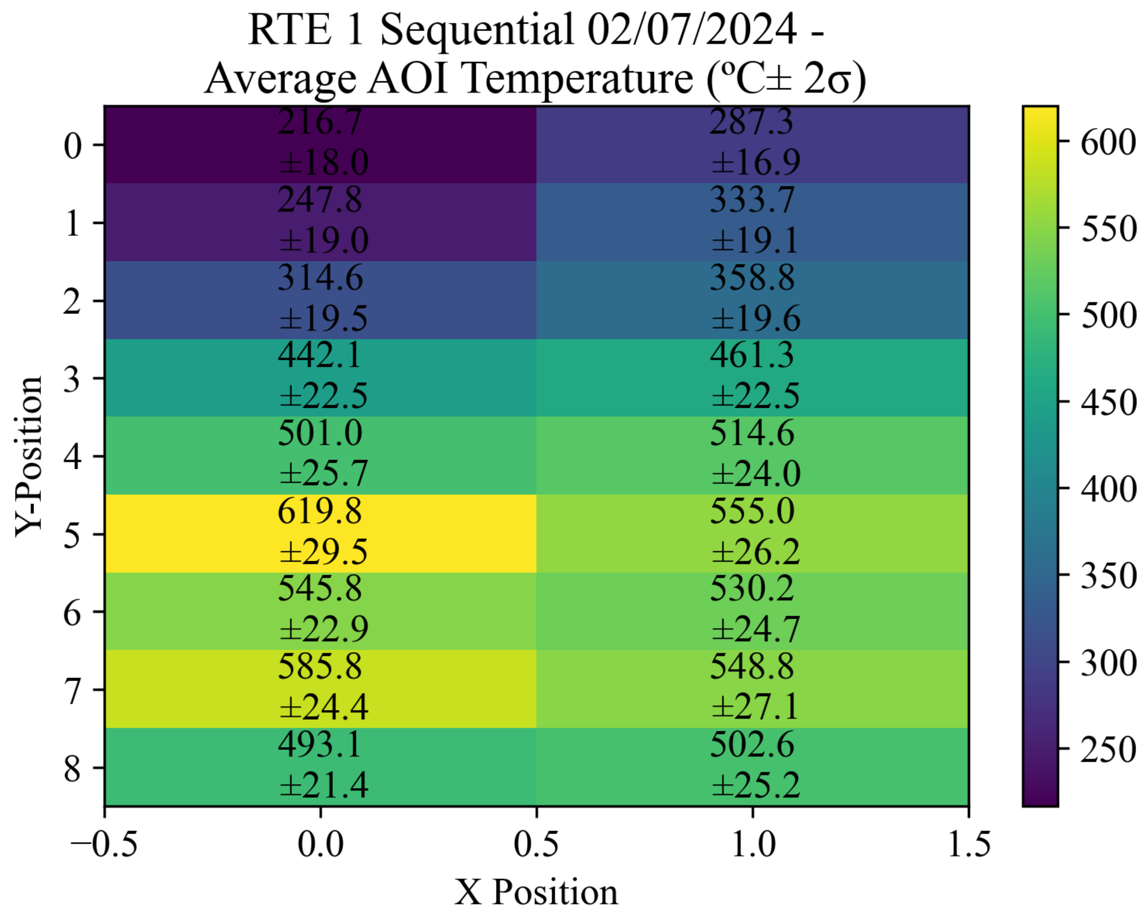


Sample failed under sequential loading at high T.

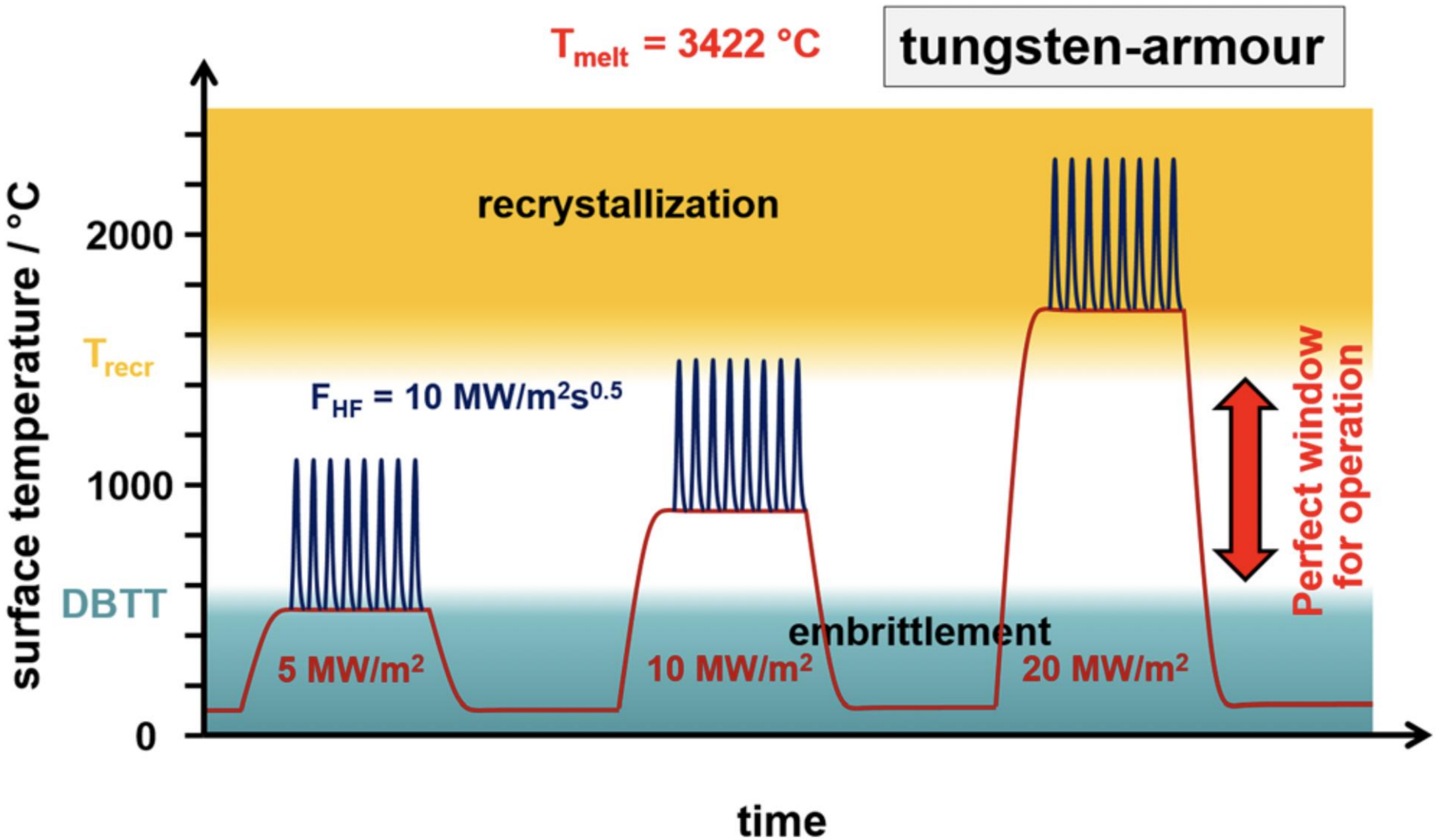


Paint speckle may affect emissivity of the sample.



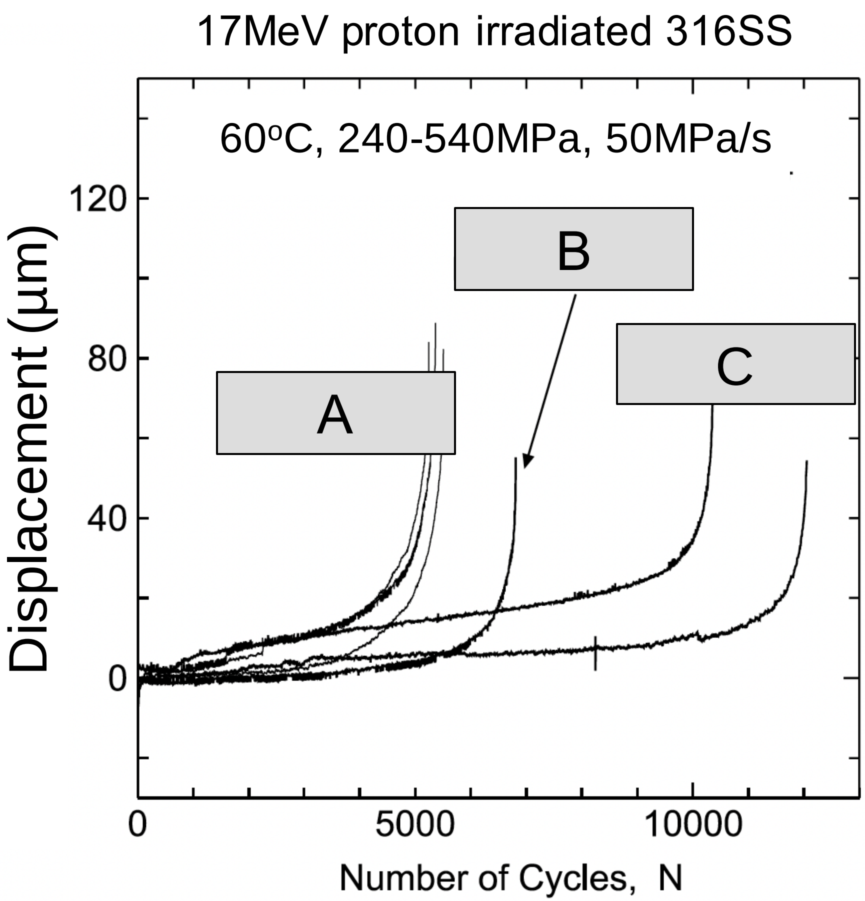


Fusion heat loads & thermal stresses will be cyclical.

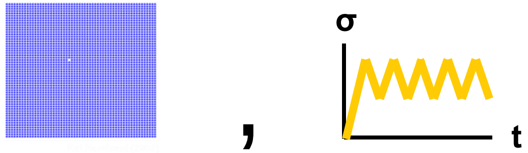


Linke et. al., Matter Radiat. Extremes 4 (2019) 056201
Rieth et al., J. Nucl. Mater. 519 (2019) 334-368

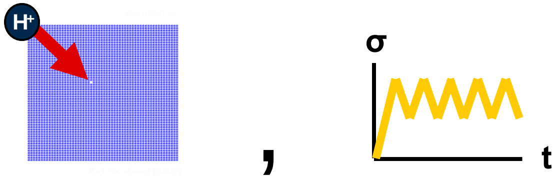
Nuclear materials' properties depend on the precise combination of irradiation, temperature, and load.



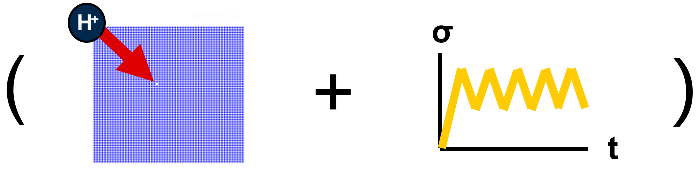
1. Unirradiated.



2. Tested post-irradiation.

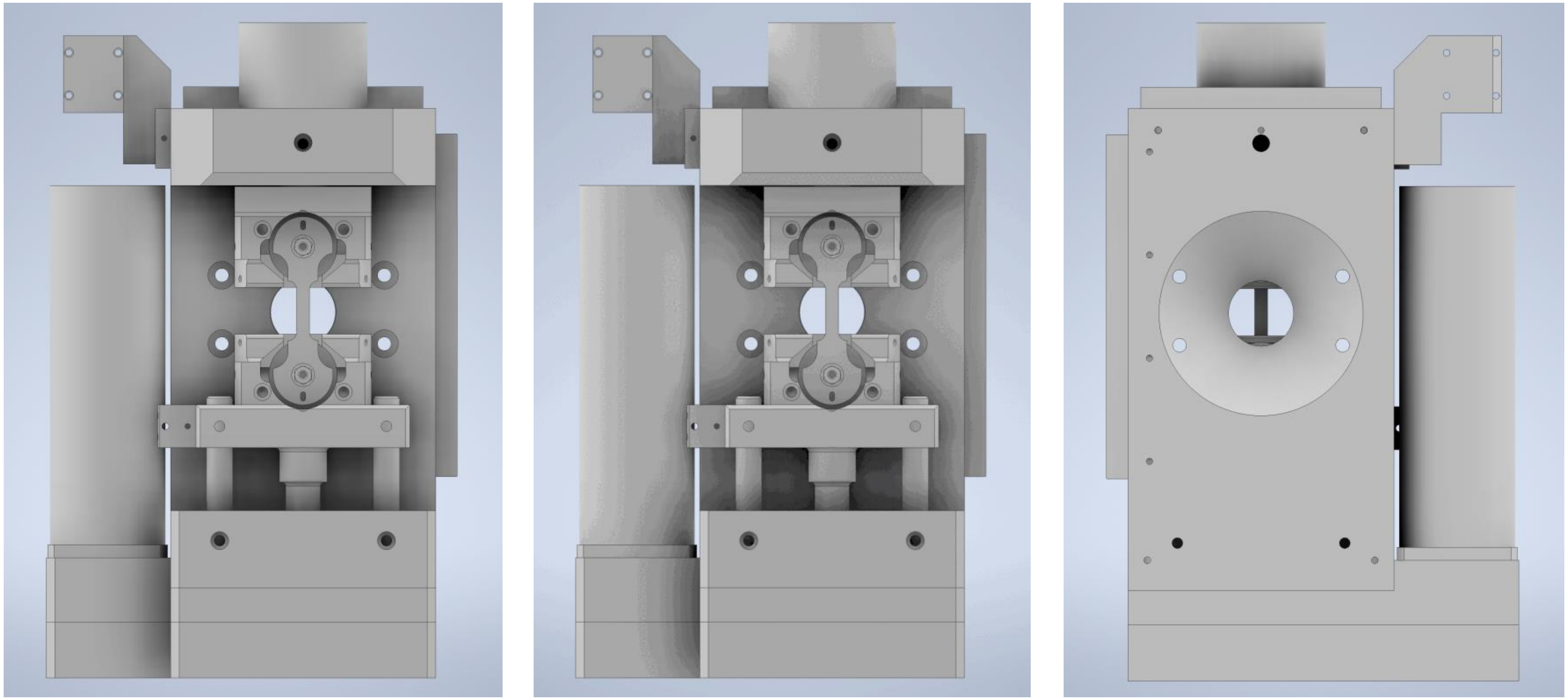


3. Tested under irradiation.

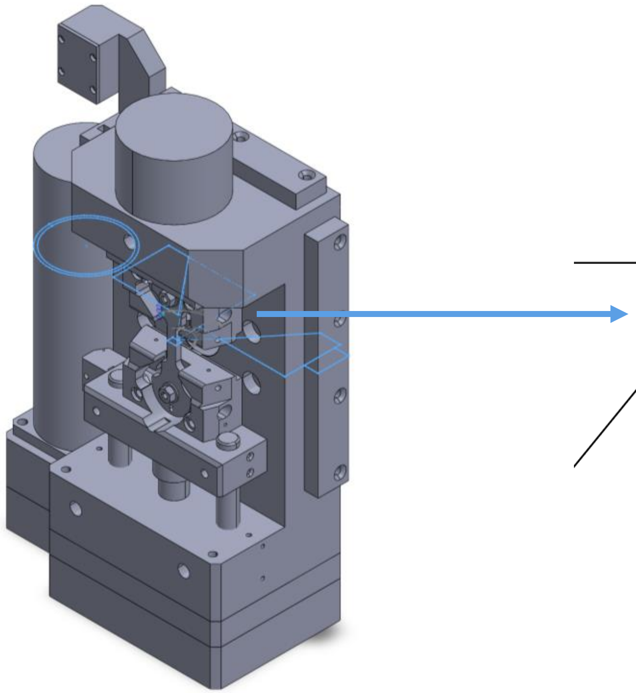


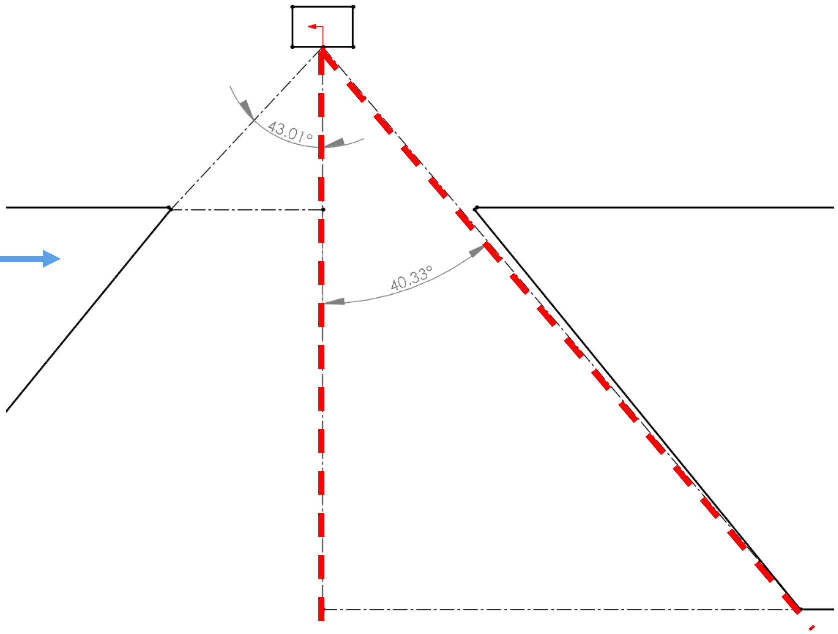
Murase et al. J. Nucl. Mater. 302 (2002) 211-216

NewTec MT1000 tensile rig

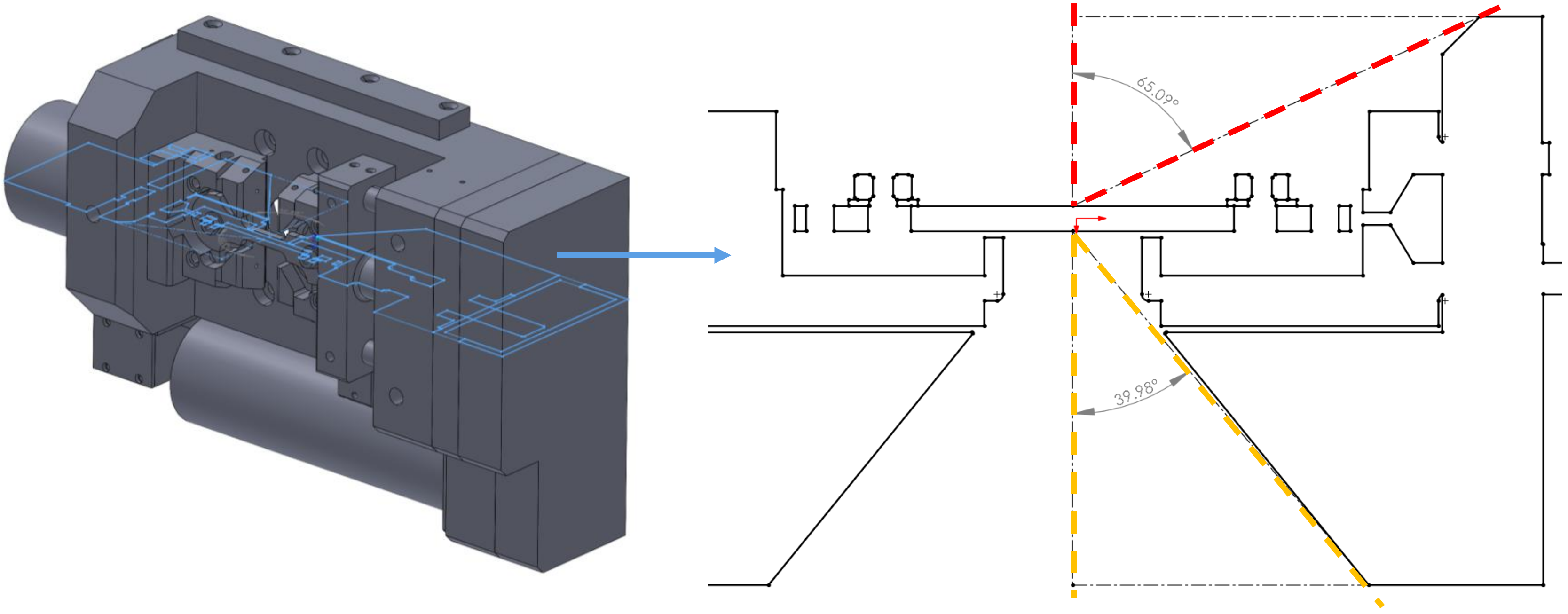


Constraints – FOV in Vertical Loading

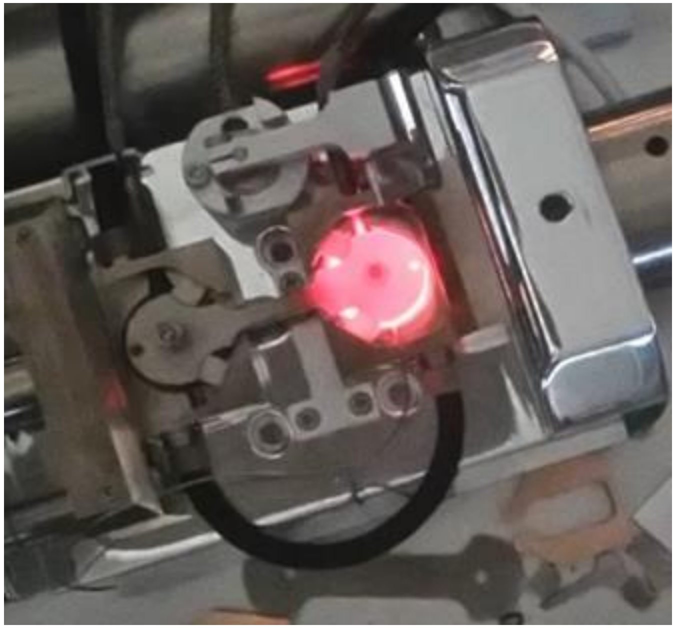


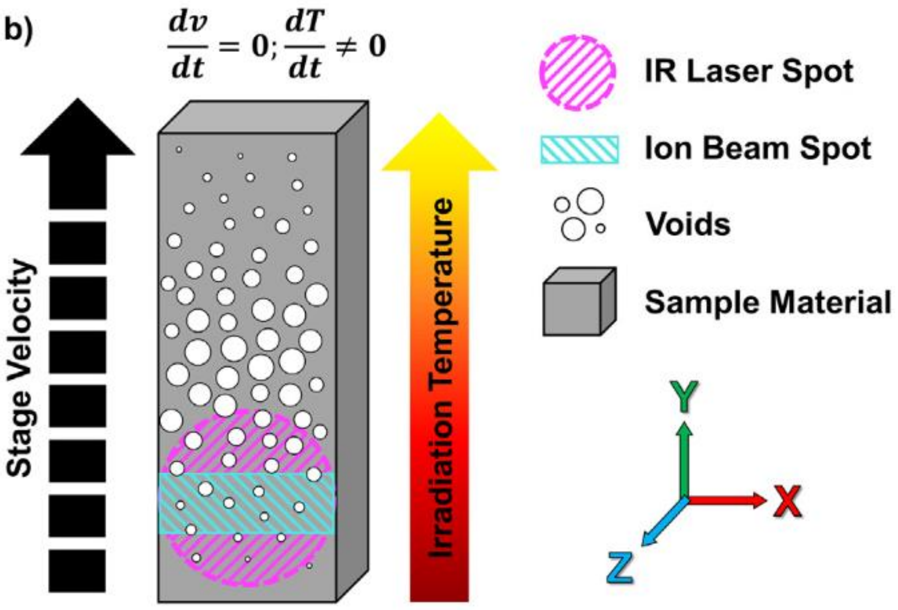


Constraints – FOV in Horizontal Loading



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





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Moorehead et al. Nuclear Inst. and Methods in Physics Research, A 1020 (2021) 165892