July 9, 2025

NSUF Workshop:

Foundations of Irradiation Testing: A Workshop for Researchers

INL/MIS-25-85883

Mechanical and Environmental Testing

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Motivation

- Need for structural materials and fuels for advanced reactor systems[1, 2, 3]:
  - Higher Energy Neutron Spectrum
  - Higher Temperatures
  - Corrosive Environments
- Example: research on new materials is being conducted, including on Multi-Principle-Element-Alloys (MPEA's, aka High-Entropy-Alloys, aka Complex-Concentrated-Alloys)
  - In a 2021 review paper[4], ~4000 papers published on MPEA
  - Of those, only ~100 were focused on nuclear applications and were topical on fabrication, modeling, ion irradiation, and corrosion.
  - Very limited neutron irradiation data on MPEAs
  - Nearly limitless composition space for MPEAs

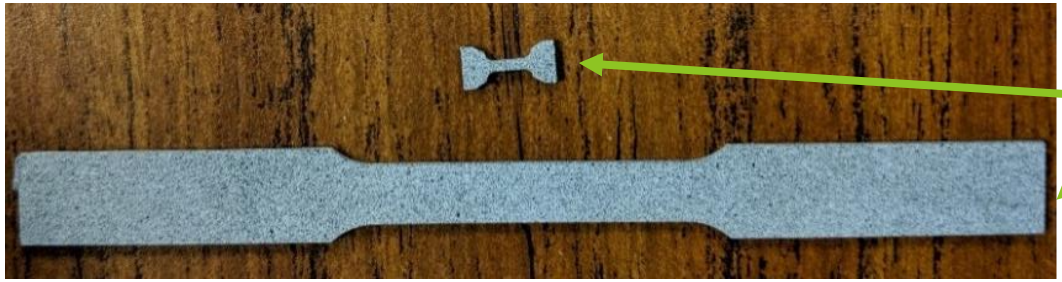
- This talk will focus on mechanical testing, but other kinds of testing such as creep, stress corrosion cracking, etc are possible.

1 – https://doi.org/10.1016/j.matre.2021.01.002
2 – https://doi.org/10.1016/j.cossms.2016.10.004
3 – https://doi.org/10.1038/ncomms13564
4 – https://doi.org/10.3390/e23010098

Challenges and Opportunities

- How to reduce time and cost to perform neutron irradiation of structural materials?
  - Develop ”Standard Capsule” with analysis basis to perform irradiations in ATR “A” positions. Reduces cost and time to get new experiments into ATR using highly available “A” positions.
  - Allows for large numbers of specimens to be irradiated.
- How to deal with large numbers of specimen materials?
  - reduce specimen size to accommodate greater number of specimens in reactor
    - Validation of smaller specimen size for mechanical testing[1]

1 – Karnati, Isanaka, Zhang, Liou, Schulthess, “A Comparative Study on Representativeness and Stochastic Efficacy of Miniature Tensile Specimen Testing”, Accepted.



Miniature “MT2” Tensile Specimen
ASTM E8 Standard ”Sub-sized Specimen”

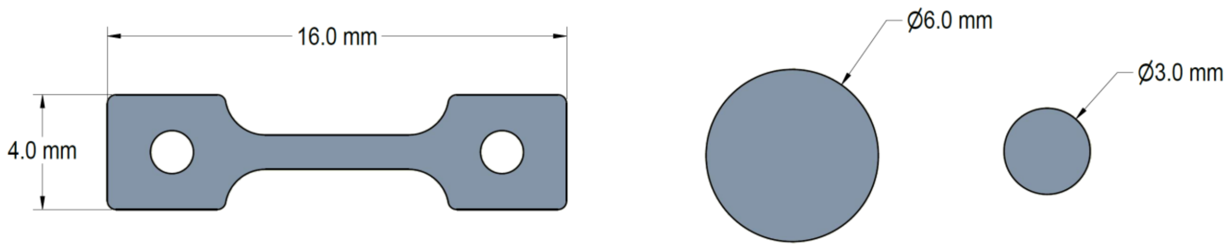
Challenges and Opportunities

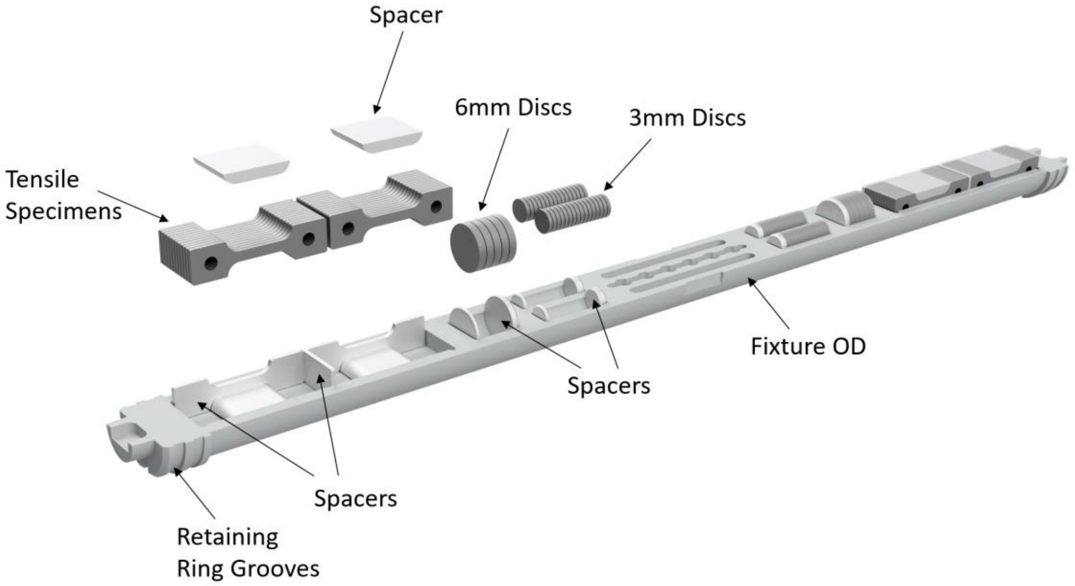
- How to increase throughput, reduce cost, and increase data extracted from each specimen?
  - As of 8 July 2022, 1,683 entries in the NSUF Material Library containing the word “Tensile” in the sample description
  - Automate mechanical testing,
  - Incorporate advanced Digital Image Correlation Analysis
    - Localized Strain Mapping
    - Spatial variation
    - Inhomogeneous materials
    - Etc.

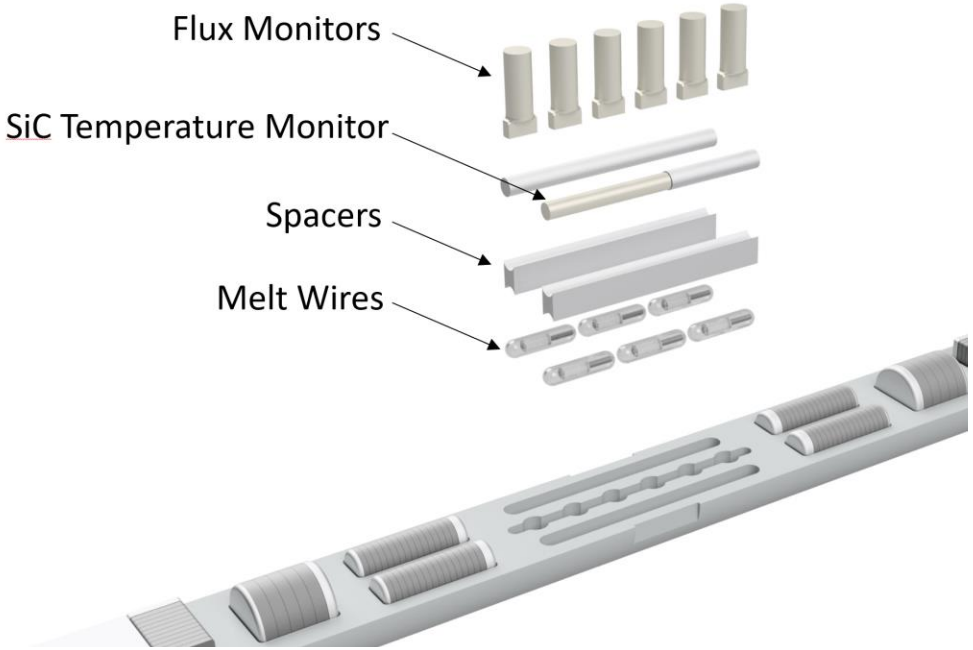
TTUSC – Tensile Test Using Standard Capsule

- Capsule design driven by three objectives:
  - Capsule should fit into a 15.9 mm diameter or larger position
    - Opens virtually every irradiation position in ATR (availability) and allows selection of test position based on flux values without limitation of the vehicle
  - Capsule should accommodate and maximize the amount of standard specimen sizes used for structural materials irradiation testing
    - Basketless design with interlocking endcaps increases inner diameter (more room for specimens) and makes reconfiguration between cycles possible.
  - Capsule analysis should be enveloping in nature so future tests can leverage the analysis to decrease deployment time
    - Parametric analysis model developed using COMSOL to allow rapid analysis for future tests.

TTUSC - Design



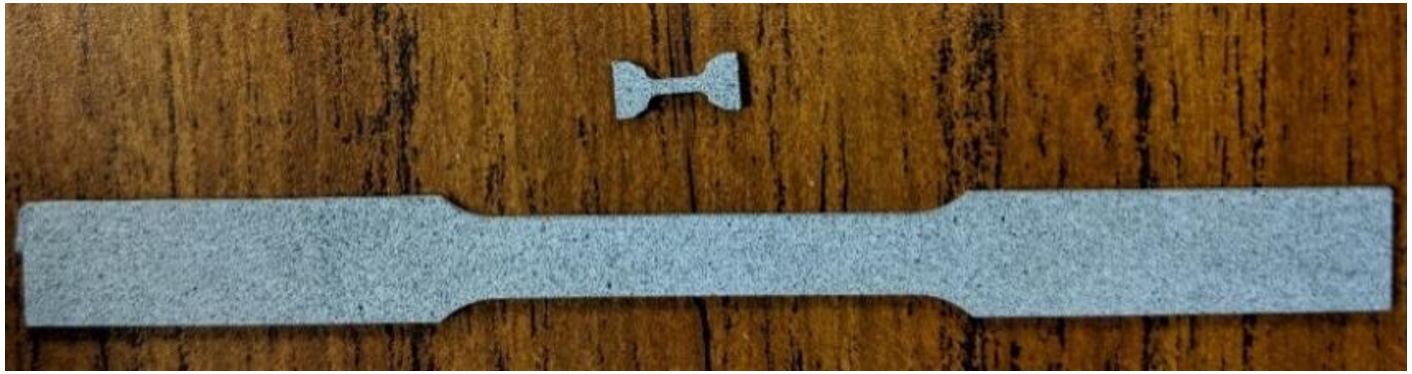




Successfully deployed for different irradiation testing campaigns:

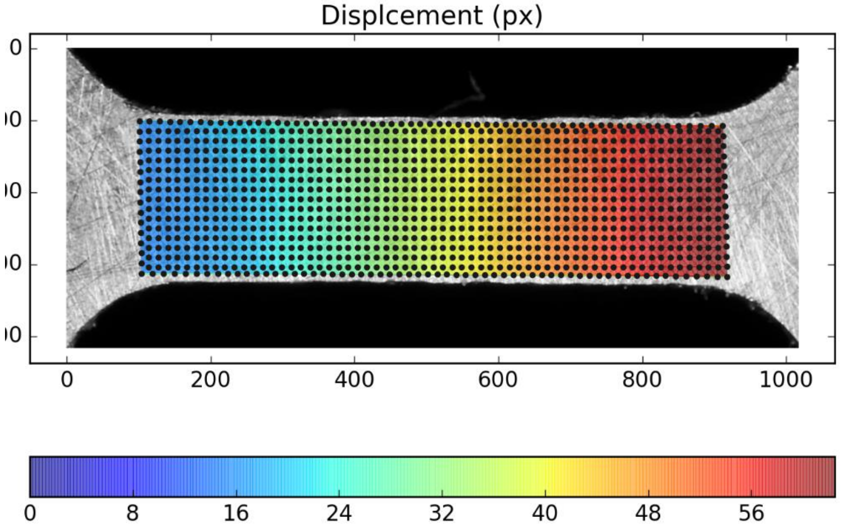
1) TTUSC
2) SAM-2
3) SAM-3
4) GENIE

Miniature tensile specimen



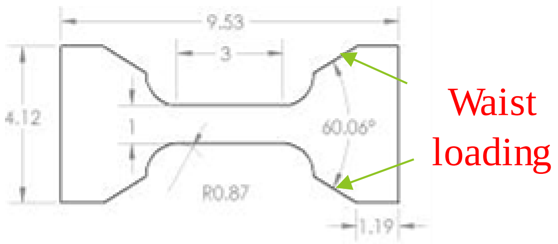
- Material characterization
- Chemistry variation
- Spatial variation
- Process variation
- Benchmarking
- DIC analysis

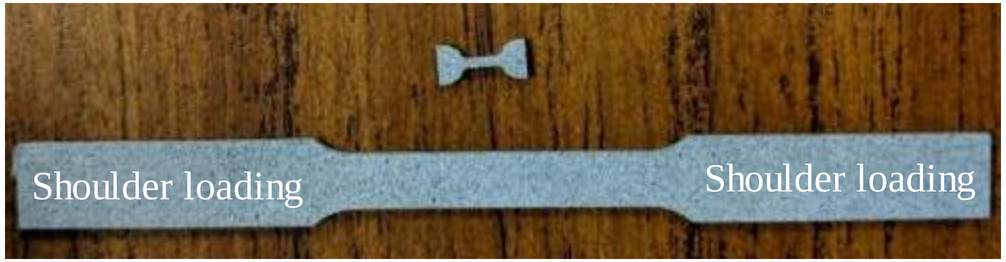
- ASTM sub-size “dog bone” specimen
  - 102 mm long with a gage length of 25.4 mm
- Miniature specimen
  - 9.65mm long with a three mm gage length

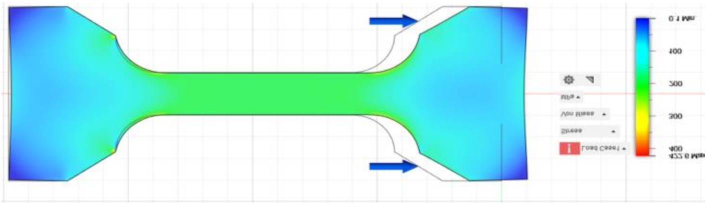


Considerations of specimen design

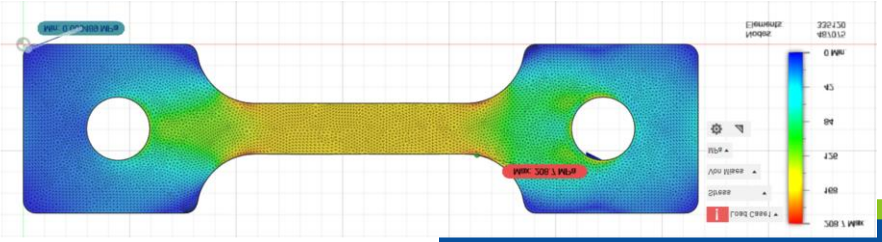
- What we call "waist loading" to minimize complexity of miniature specimen
  - Minimizing manufacturing complexity for tough and/or brittle materials
  - Minimizing testing complexity due to the load applications without the need for clamping, pin loading and other loading mechanisms (i.e. easier for remote work in hot cell application)
  - Minimize stress concentrations





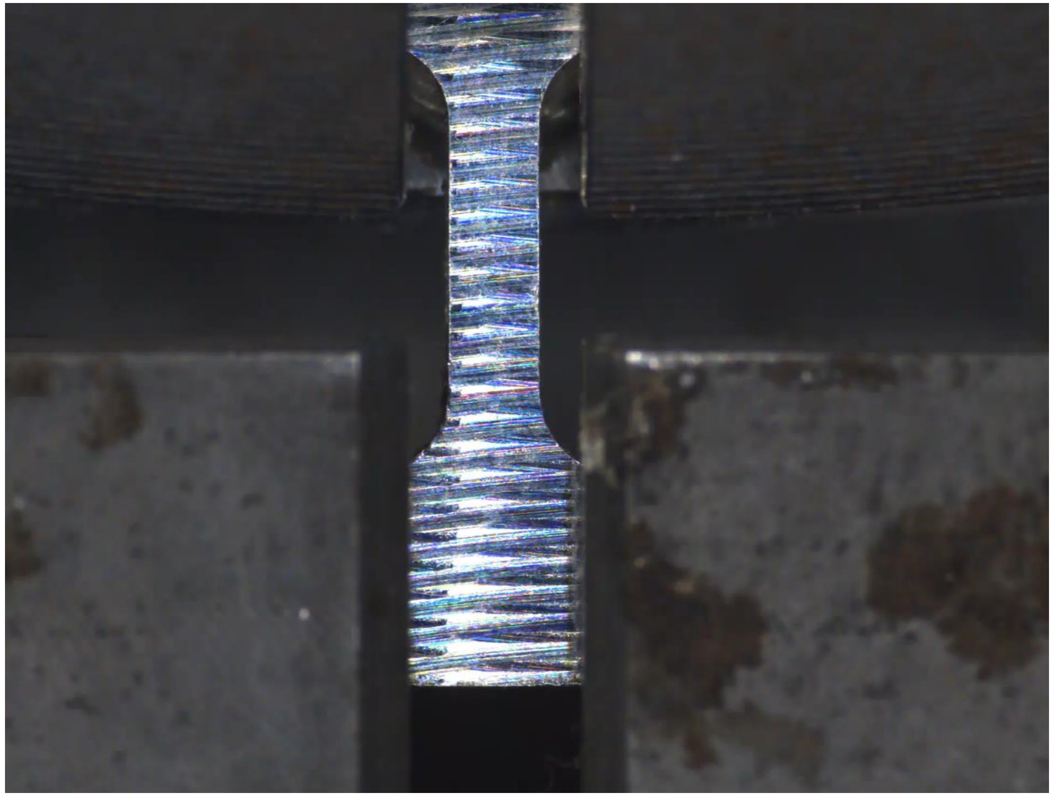


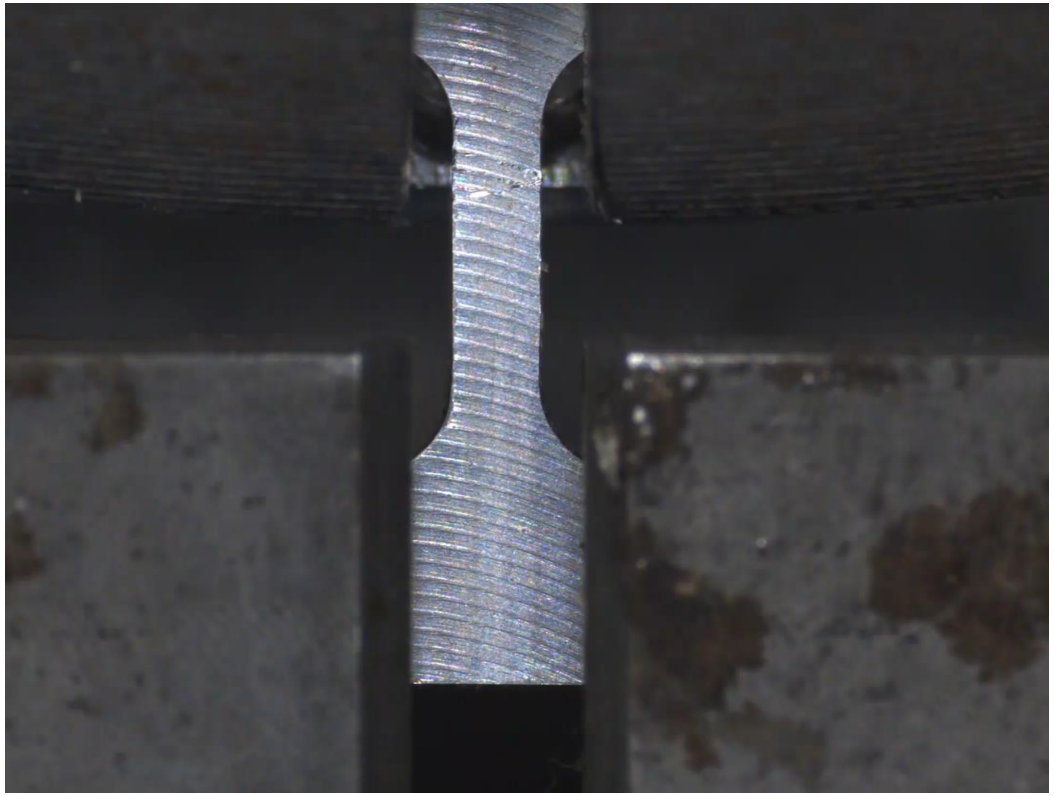
MT2



SSJ-3

Effects of manufacturing process





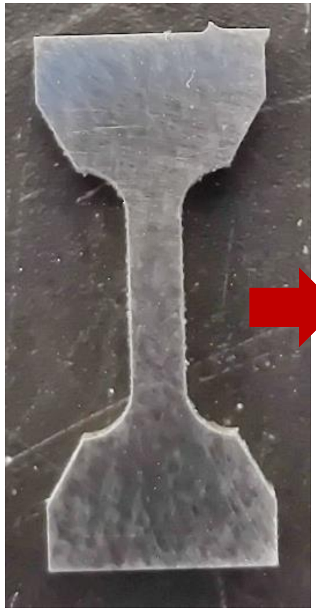
- CNC machining tends to work harden material
- Leading to zones that affect the neck formation and material elongation behavior

Specimen preparation

- Miniature specimen come with manufacturing and specimen preparation challenges
- EDM Manufacturing process chosen to minimize work hardening
- Lapping process included to remove scale, oxide layer and other artifacts like burr, formed during specimen manufacture

EDM specimen before polishing using 600 grit and 800 grit media. Average of 3 measured values for the dimensions

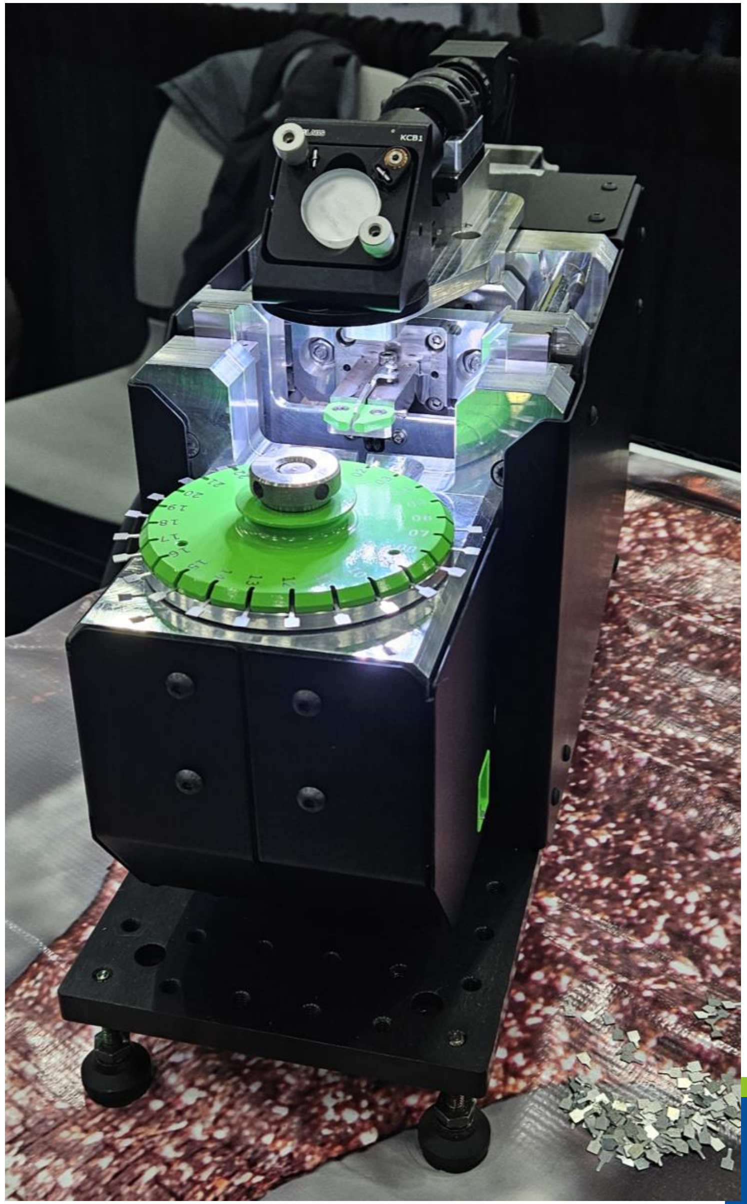
Length = 10.06 mm
Width = 0.91 mm
Thickness = 1.01 mm



EDM specimen after polishing using 600 grit and 800 grit media. Average of 3 measured values for the dimensions

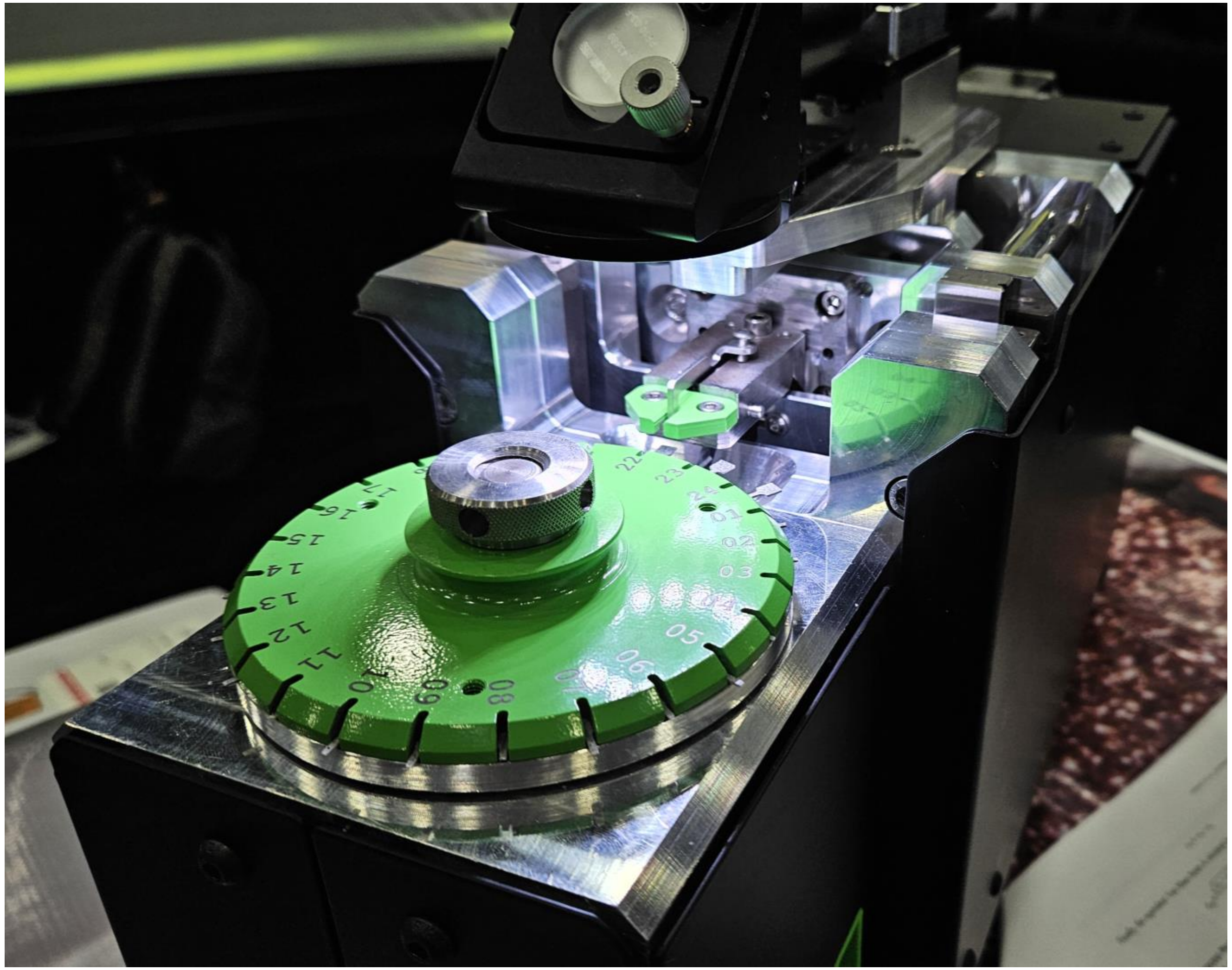
Length = 10.06 mm
Width = 0.91 mm
Thickness = 0.97 mm

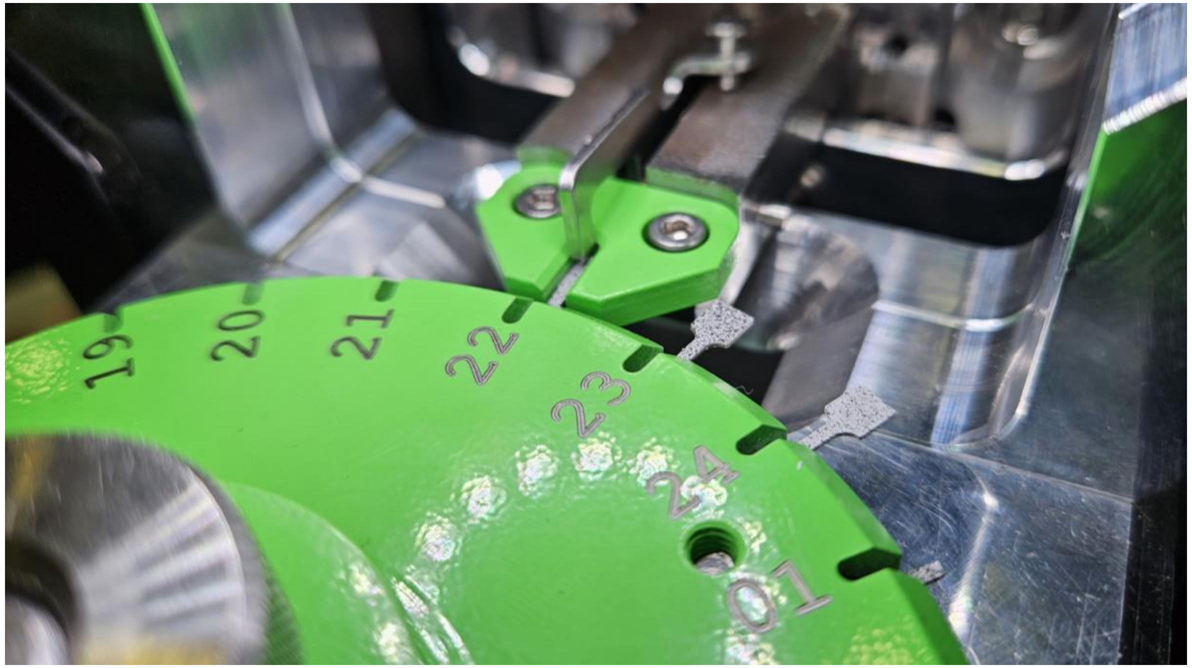
Advanced Mechanical Test System (AMTS), non-rad



- Cartridge design to house 24 specimen
- Rotational indexing to position specimen, apply preload, and test

Advanced Mechanical Test System (AMTS)





- Close ups of AMTS performing tests
- SSJ Specimen or other specimen geometry like MT2
- Speckle pattern applied

Data generated

PINE PRODUCT INNOVATION AND ENGINEERING

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Date: August 20, 2024
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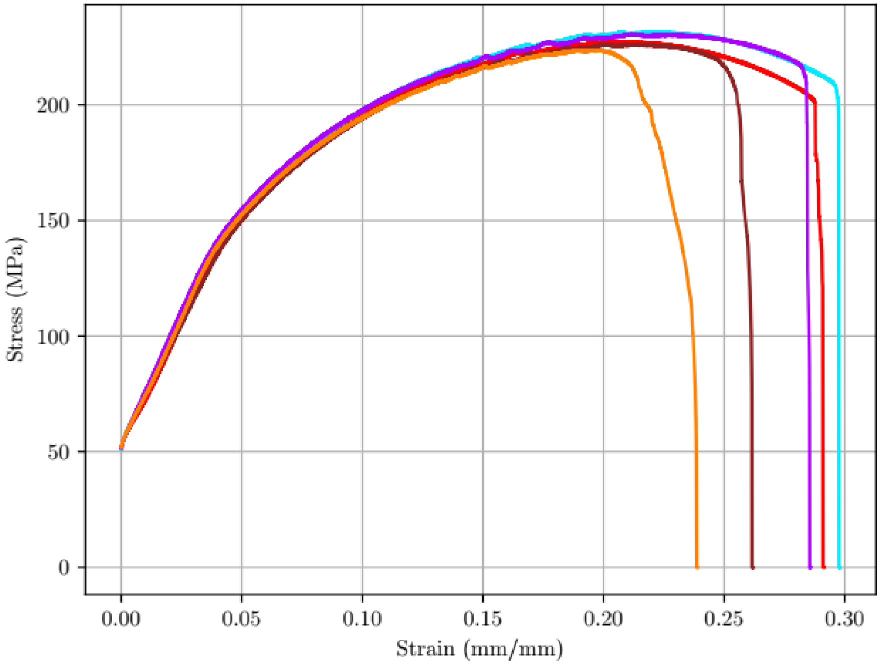
Tensile Test Report

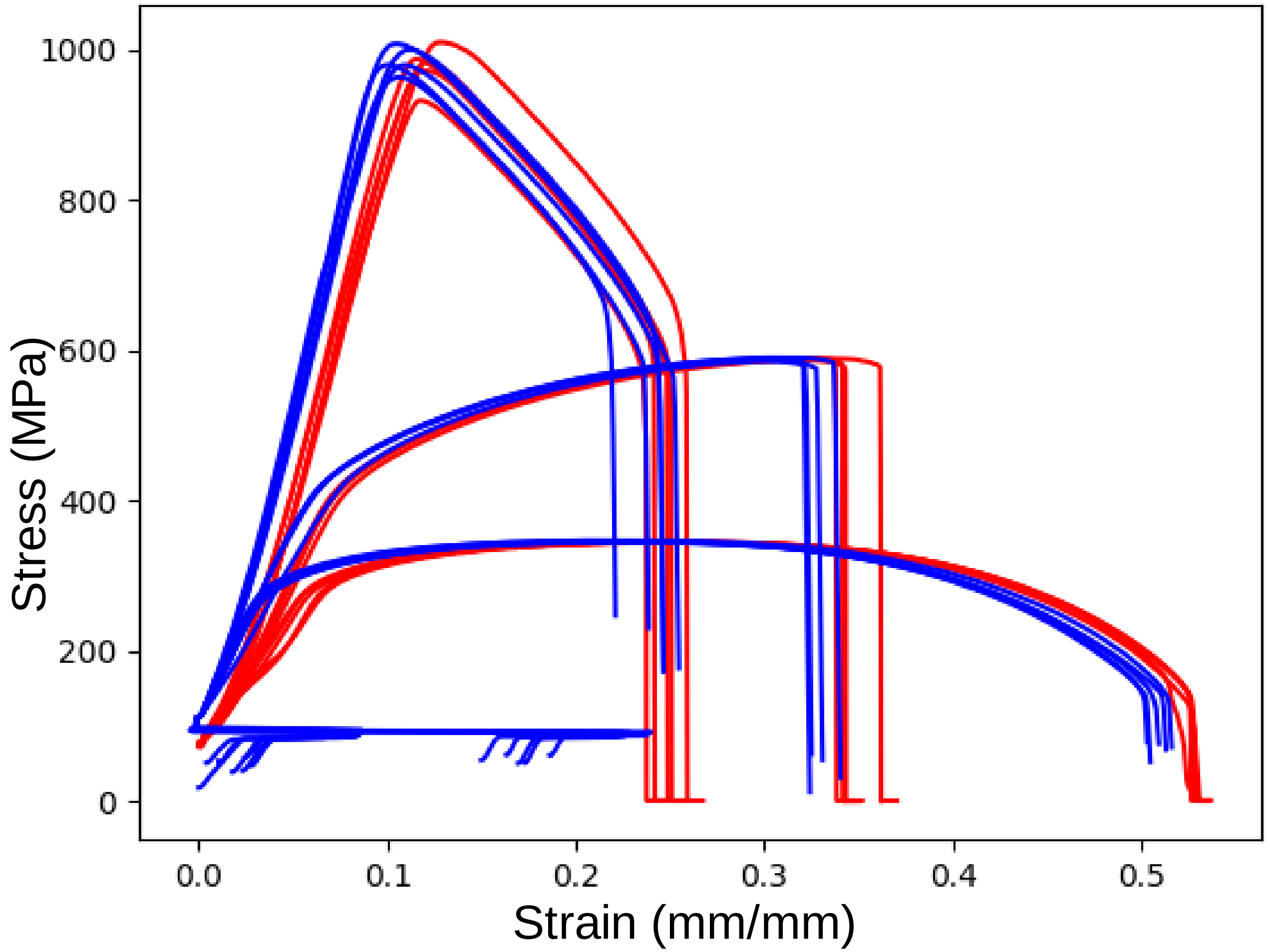


Sample Name	UTS (MPa)	0.2% YS (MPa)	Break Strain (mm/mm)
2E1	227.35	156.67	0.291
2E2	226.30	156.40	0.262
2E3	231.63	159.55	0.298
2E4	230.73	158.77	0.286
2E5	223.71	153.89	0.239

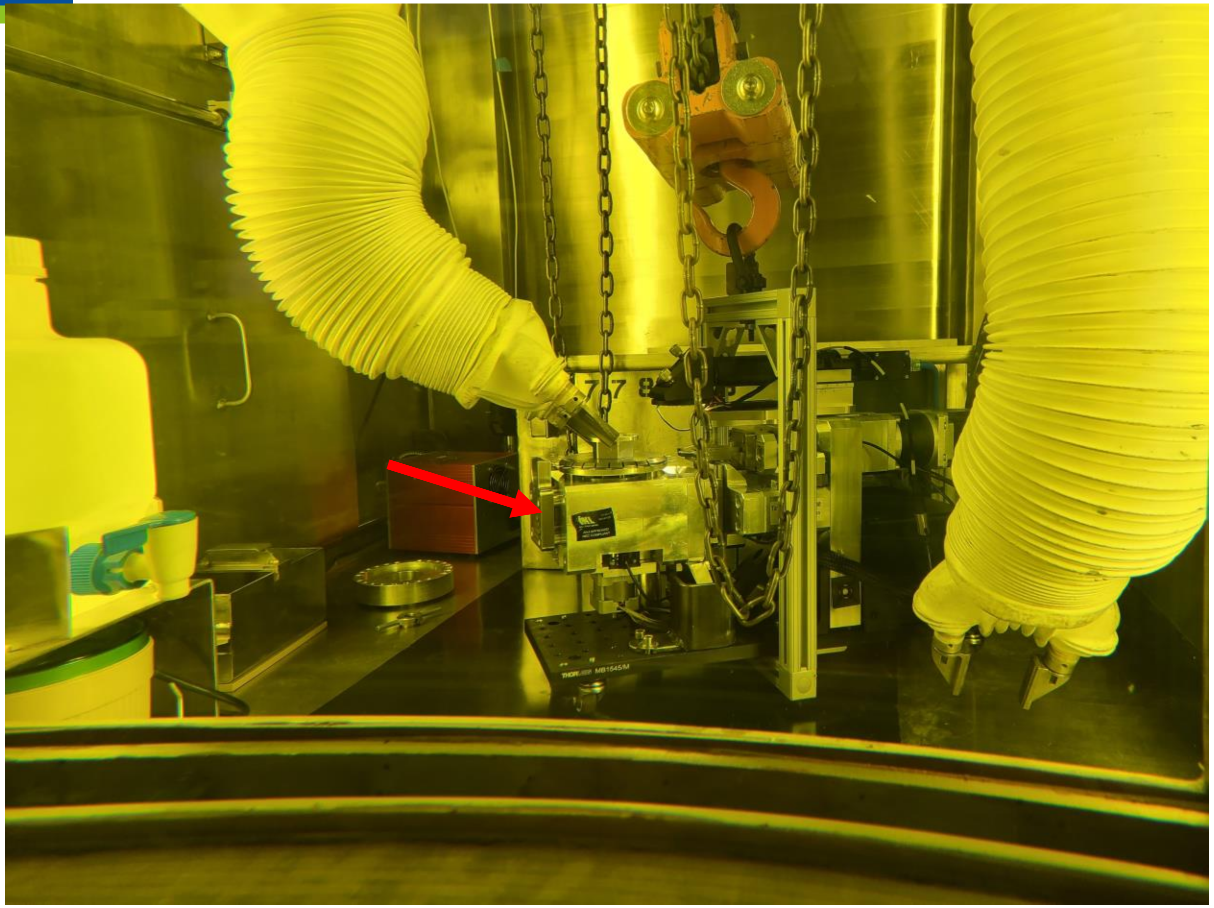
- Reports compiled for individual tests and groups with important metrics

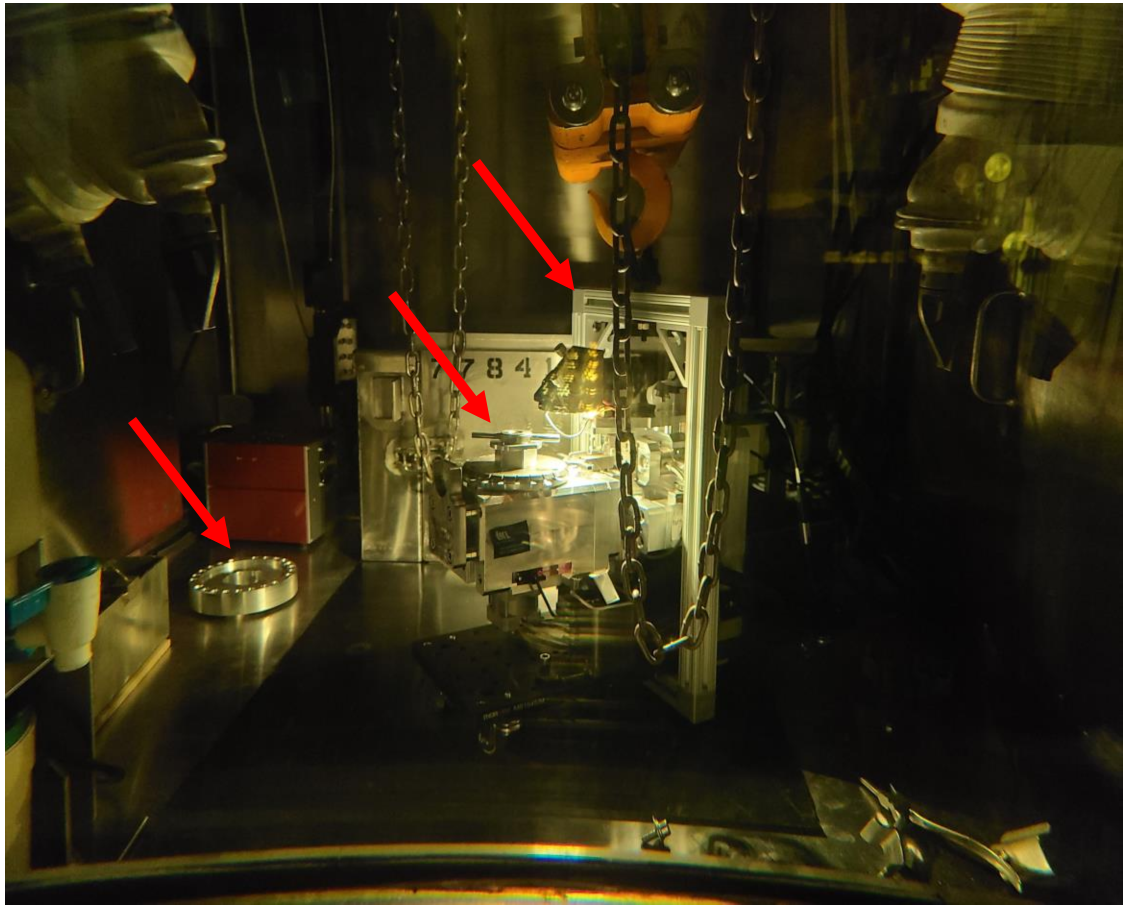
AMTS comparison with traditional test frames

- Comparative test between Tinius Olsen 25 kN frame and AMTS
- 3 materials
  - 17-4 PH steel
  - Al 7075-T6
  - C1008 steel
- 5 specimen per material each tested on the TO and the AMTS
- TO data in blue, AMTS data in red



AMTS for irradiated material, located at INL, in FASB Hot Cell

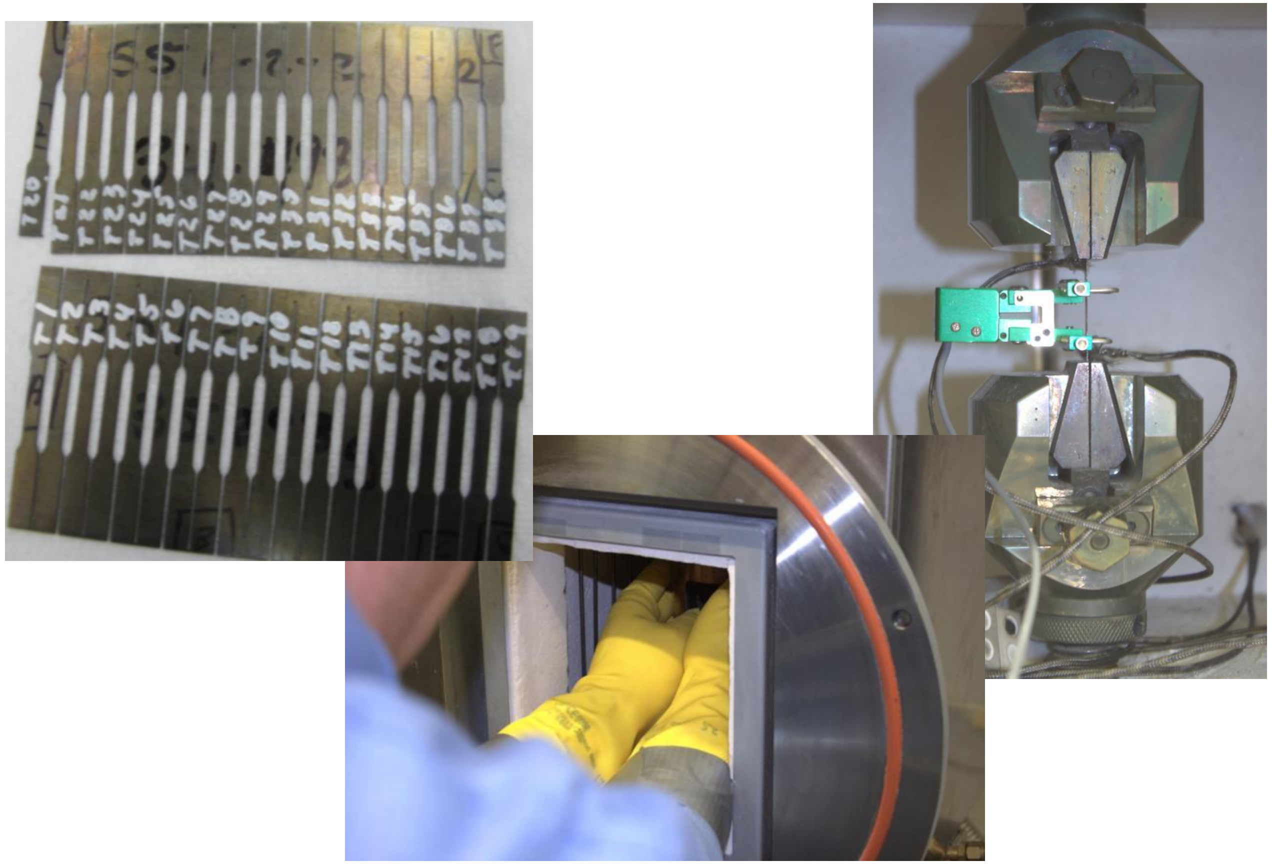


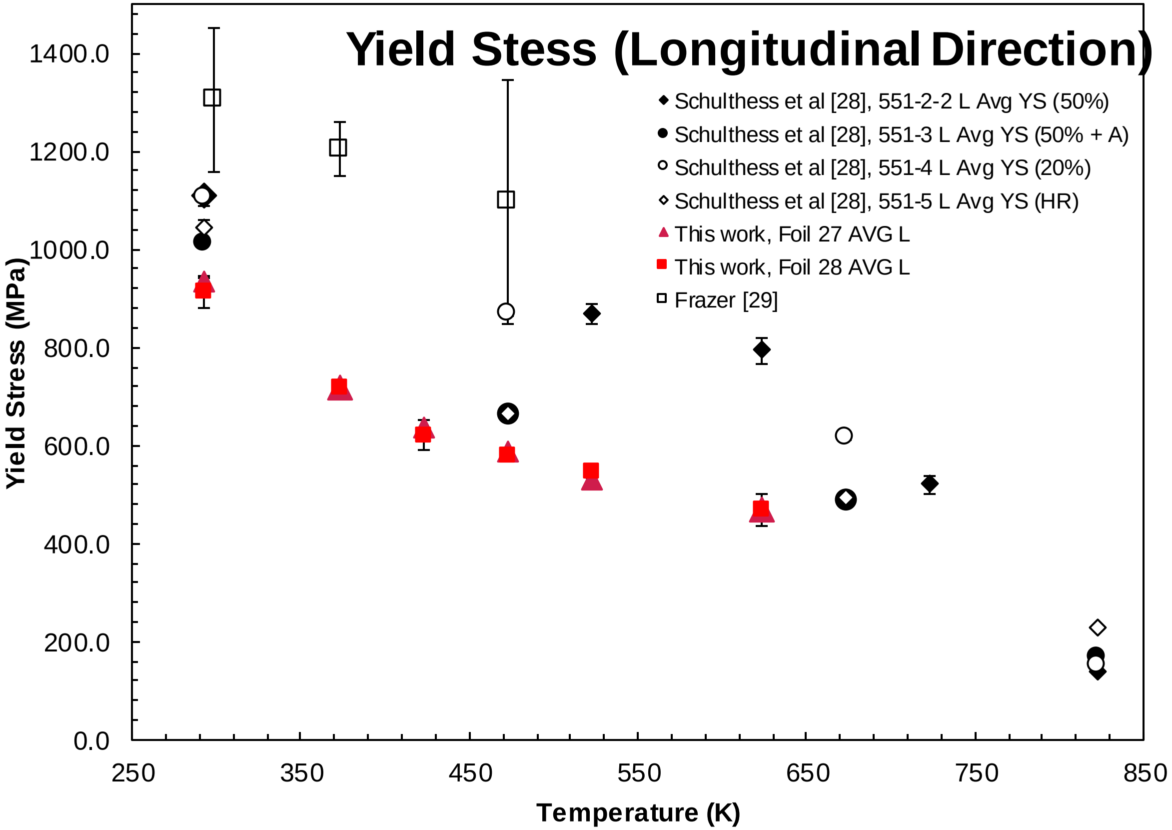


- Advanced mechanical test system installed in hot cell facility at INL, FASB
- Frame for lifting, Cartridge modifications for ease of loading
- Calibration fixture to periodically test and correct load cell

Mechanical Testing Nuclear Fuel

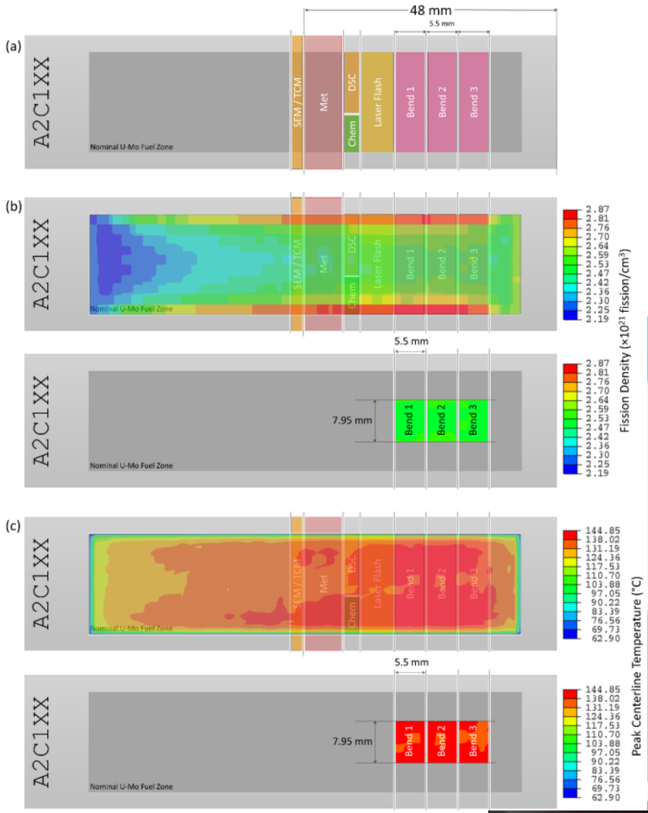
- Mechanical properties of nuclear fuel also important for understanding mechanical integrity, geometric integrity, and mechanical failure limits.
- Example of U-Mo alloy tensile tests on unirradiated material

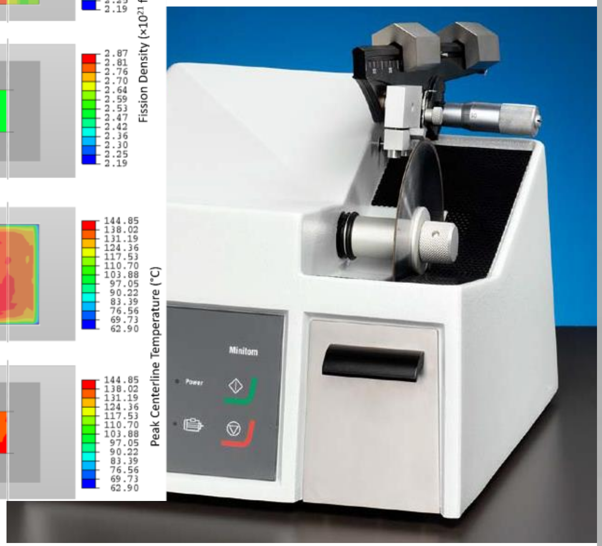


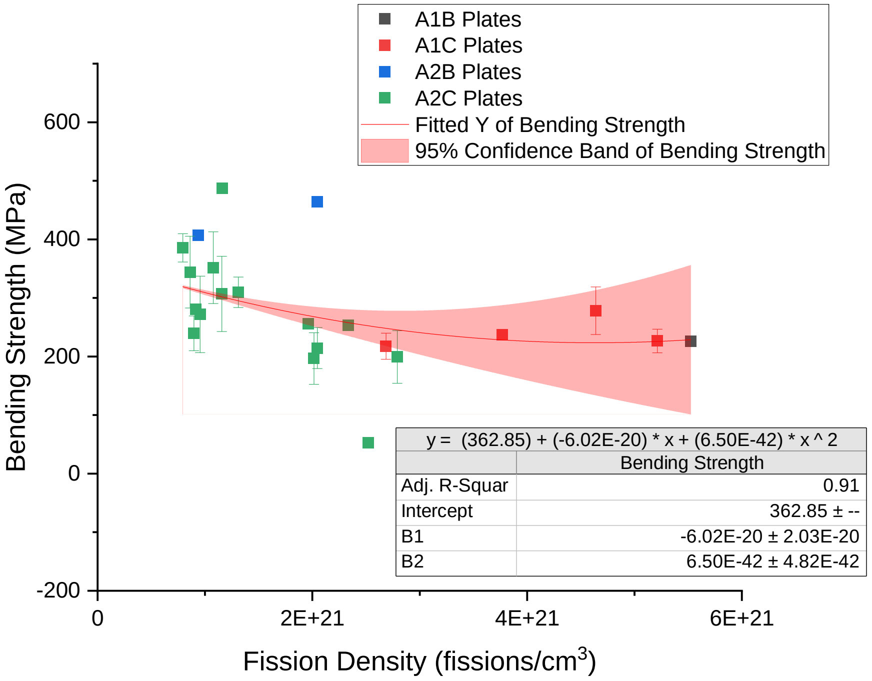


Mechanical Testing Nuclear Fuel

- 4-point bend testing irradiated U-Mo alloys
- Sample geometry (flat plates) and hot cell machining capability (no wire-EDM) means specimens are limited to rectangular geometries

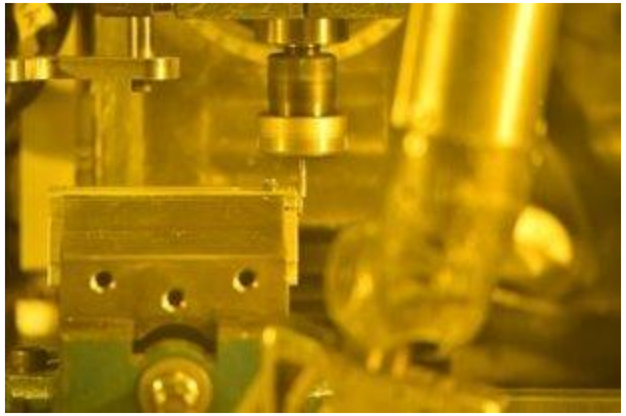


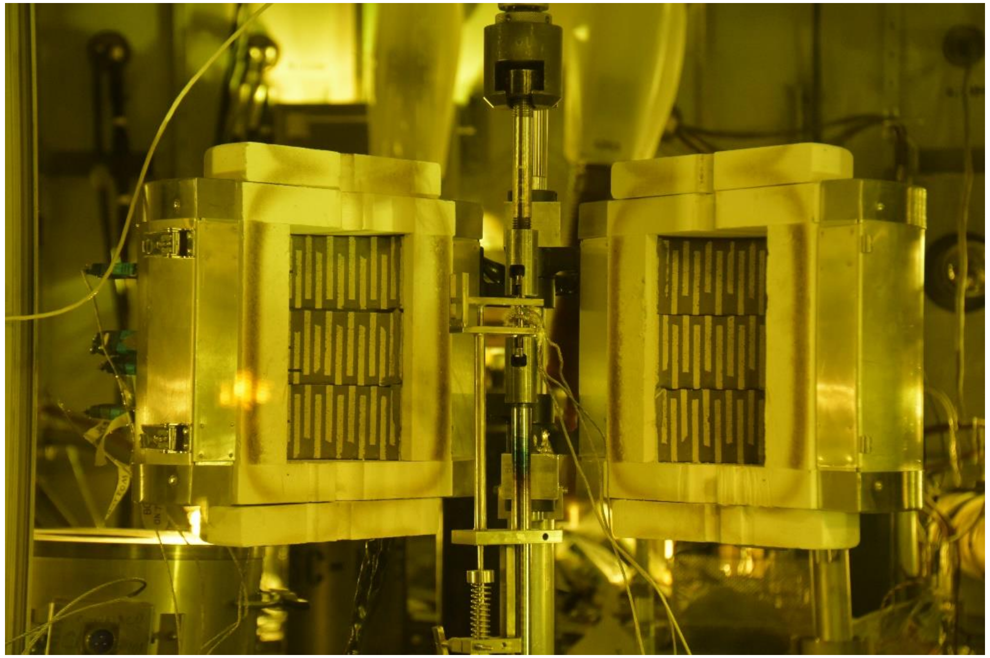




Mechanical Testing (HFEF)

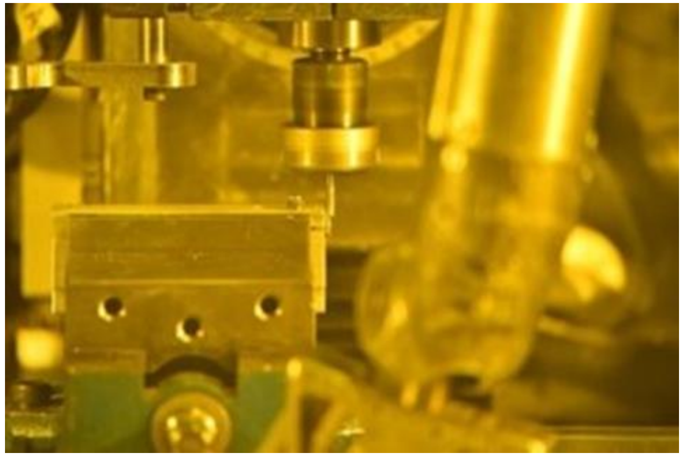
- Conventional mill used to machine test specimens
  - Customized machining jigs
- Twin column remote load frame
  - 50kN, customized for in-cell
- Clamshell furnace with pull rod feedthroughs
- Capable of many test methods and elevated temperature
  - SSJ, standard tensile, 4-pt bend, standard compression
  - Non-standard test methods possible, but require engineering effort and in-cell qualification



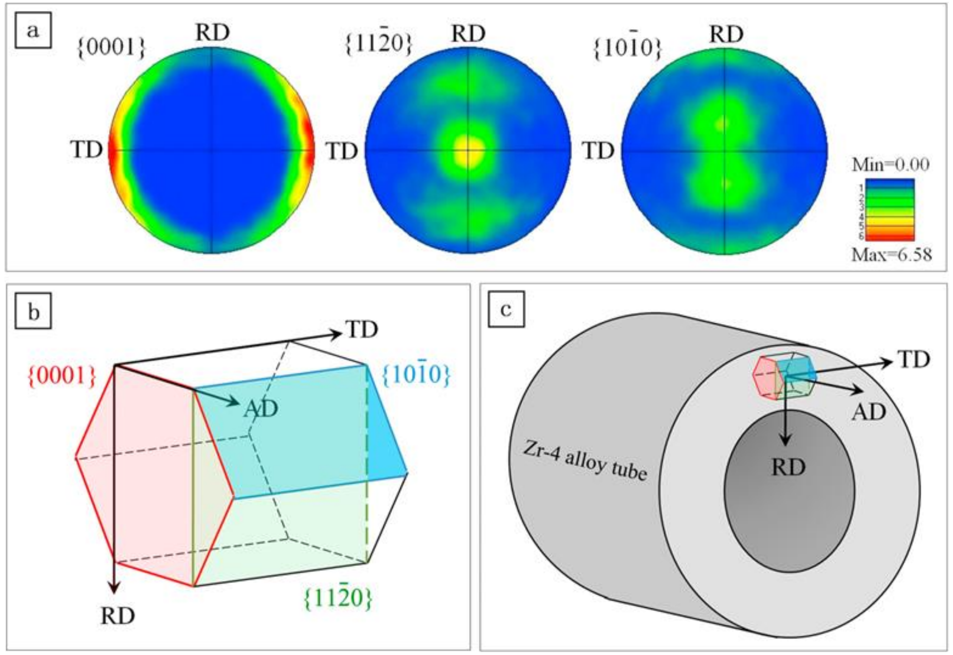


Mechanical Testing of Tube Geometries

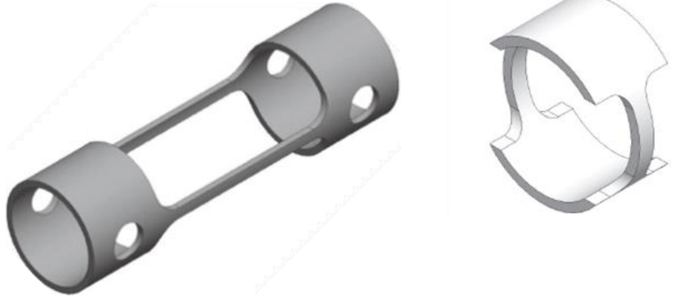
- Geometry of irradiation test samples often determines type of testing available
- Tube materials (like LWR or advanced reactor cladding) often must be irradiated in specific geometry
  - Anisotropy, coatings, oxide layers, etc.
  - No ASTM standard
  - Specimen availability limitations (maximize data)
- Ring tension, ring compression, axial tension
- Often requires machining of samples in cell
  - Can be expensive



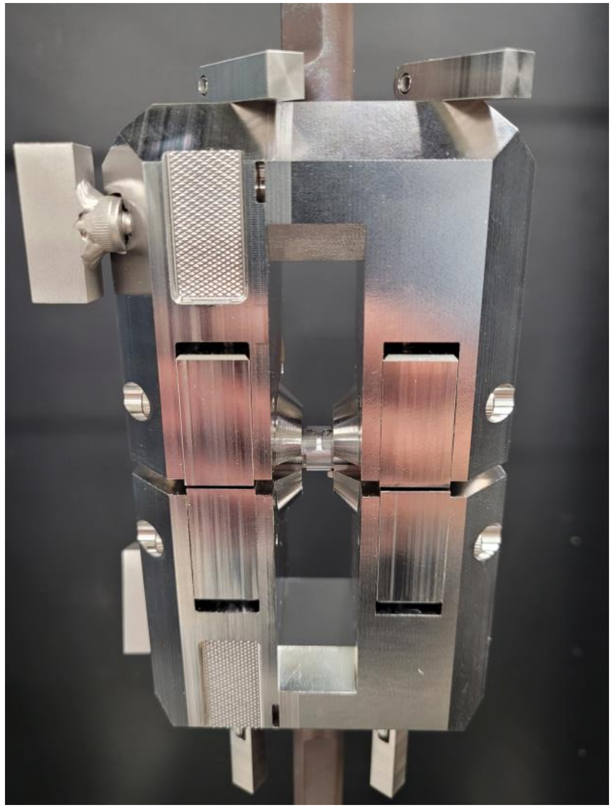
Zircaloy-4 shows strong texture, with c {0001} axis parallel to hoop or transverse direction (Liu et al., Mat. Sci. and Eng.: A, 2018)



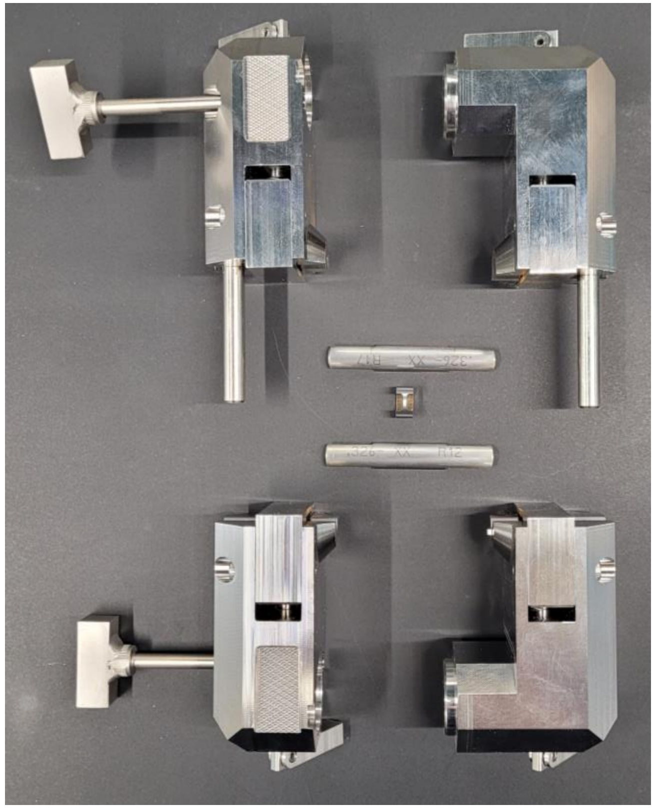
Axial (left) and hoop (right) mechanical tension tests (Kamerman et al., Nuc. Materials and Energy, 2022)



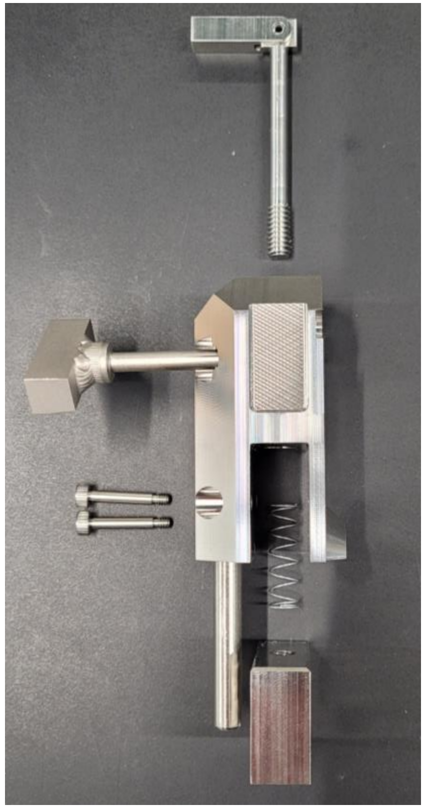
Custom Ring Tension Grip Overview



Fully assembled with ring tension specimen loaded.



Exploded view.

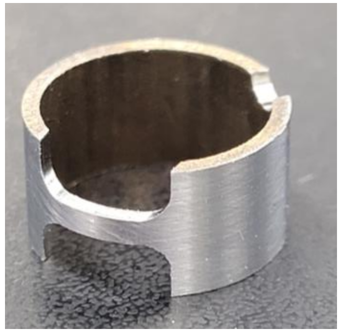


Corner exploded view.

Features

- Removable Gauge Pin Mandrels
- Adaptable Design: Cladding IDs from 0.25”-0.45”(6.35-11.43mm)
- Temperature Resistant 17-4PH H925 Construction
- Remote Operable
- Digital Image Correlation (DIC) Capable
- Ensures Ring Gauge is at 45°

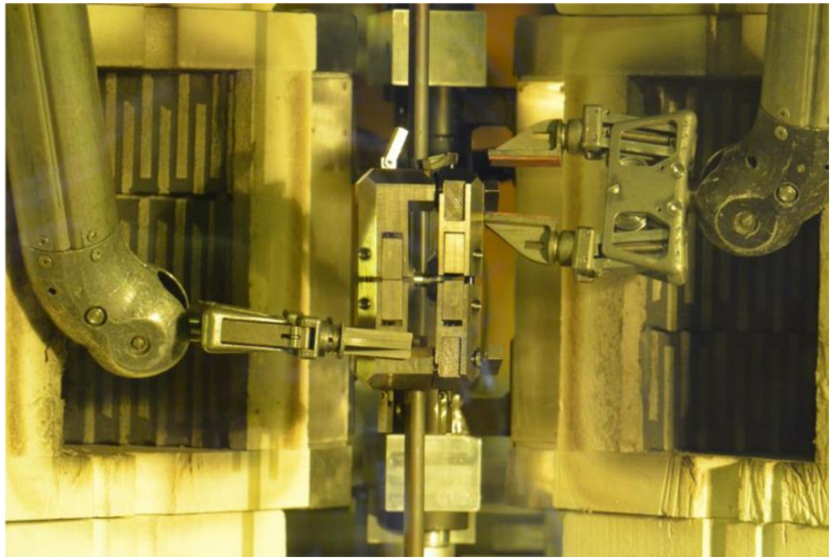
Mandrels in loading orientation with ring specimen installed.

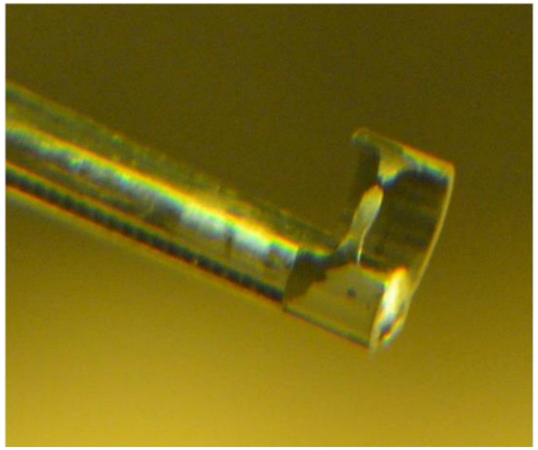


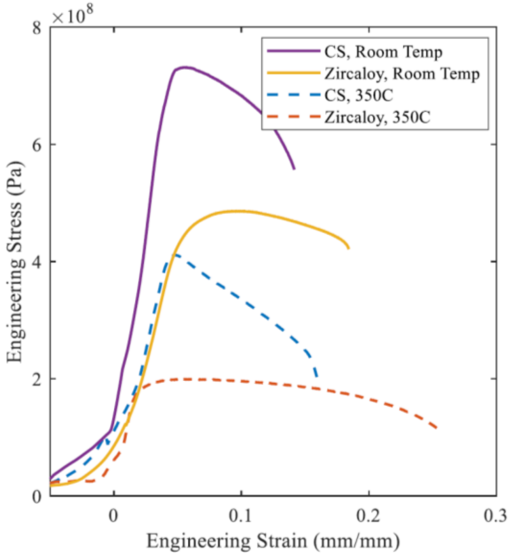
Typical ring specimen.

Petersen et al, *J. Testing and Eval.*, 2024 https://doi.org/10.1520/JTE20240106
Hansen et al, *Intl. J. Solids and Structures*, 2023 https://doi.org/10.1016/j.ijsolstr.2023.112471

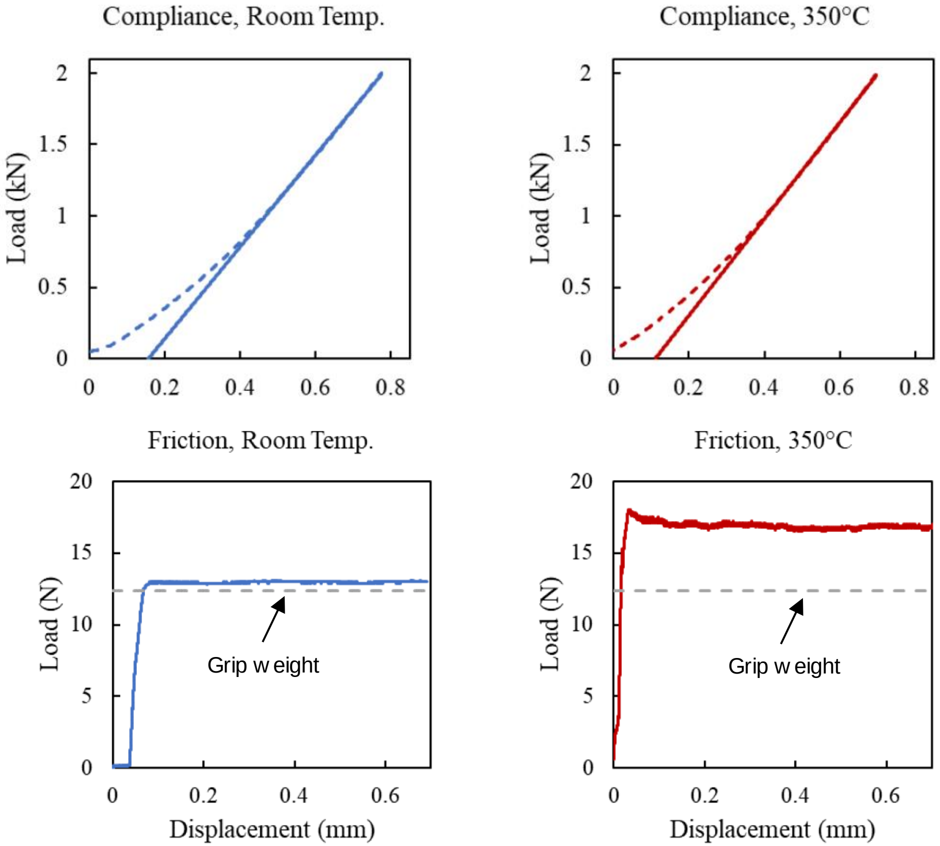
In-Cell Friction, Compliance and Ring Test







Stress-strain plots of 4 rings at room and elevated temperature.



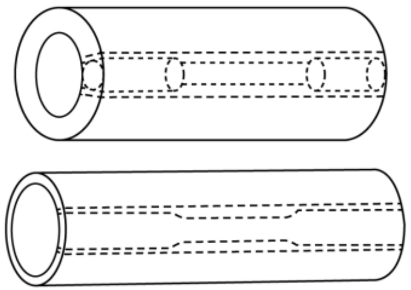
In-cell compliance and friction load-displacement curves at room and elevated temperature.

- New guide rods showed a reduction in friction.
- Sharp load drops during elevated ring tests were not present.
- Repeatability was improved by removing dependence on T-handle torque.

Robert Hansen, Phil Petersen David Kamerman, Fabiola Cappia, Katelyn Baird
Qualification of the Optimized Hemicylindrical Ring Tension Test Fixtures,
INL/RPT-23-73161, June 2023

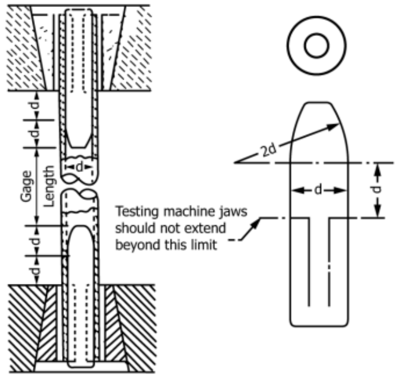
Axial Tension Test – Motivation

- Tubular materials with microstructural anisotropy
  - e.g. Zirconium alloy nuclear cladding (HCP)
  - Requires axial, hoop direction tests
- Axial Tension Test (ATT)
  - Maximize # of tests, limited material
  - Leverage existing infrastructure (load capacity)
  - Unique geometry, irradiated material constraints prevent use of ASTM E8
- Investigate ATT configurations
  a) Aligned pin loading (original)
  b) Offset pin loading
  c) Collet loading
- Compliance outside gauge an issue



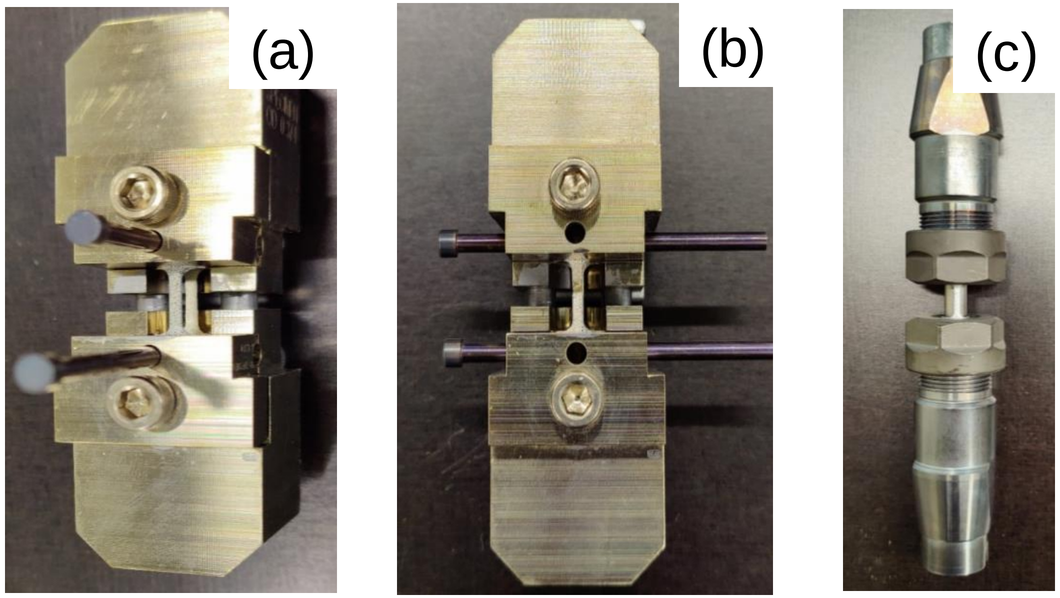
NOTE 1—The edges of the blank for the specimen shall be cut parallel to each other.

FIG. 13 Location from Which Longitudinal Tension Test Specimens Are to be Cut from Large-Diameter Tube

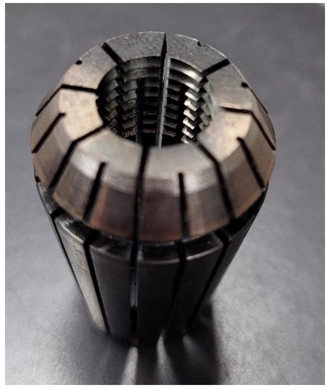


NOTE 1—The diameter of the plug shall have a slight taper from the line limiting the test machine jaws to the curved section.

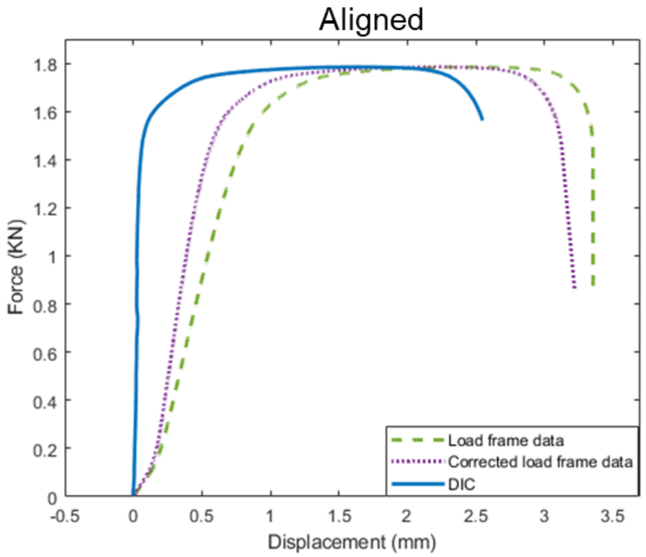
FIG. 12 Metal Plugs for Testing Tubular Specimens, Proper Location of Plugs in Specimen and of Specimen in Heads of Testing Machine

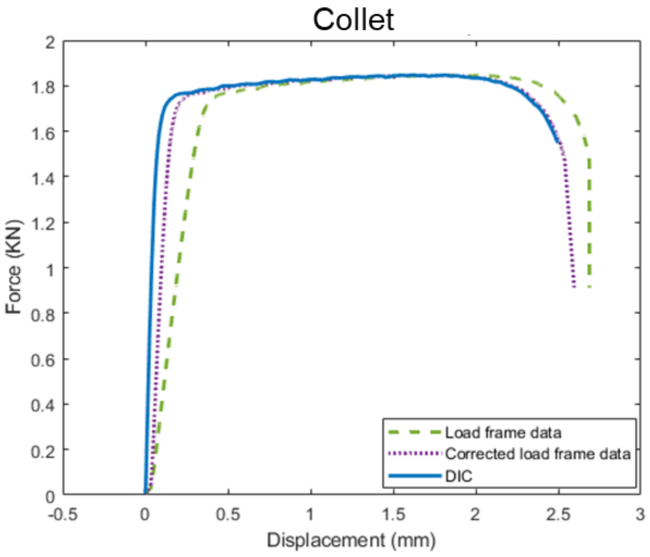


ATT – Configurations



- Aligned pin loading
  - Initial design
  - Prone to elongation in hole post-test
  - Potential stress concentration with gauge?
- Offset pin loading
  - Rotated hole position 90°
  - Reduce stress concentration
  - Potential bending in shoulders
- Collet loading
  - Avoid deformation in hole
  - Potential challenge for gripping
  - Sharp teeth machined into collet ID
  - Performed the best (greatly reduced compliance)



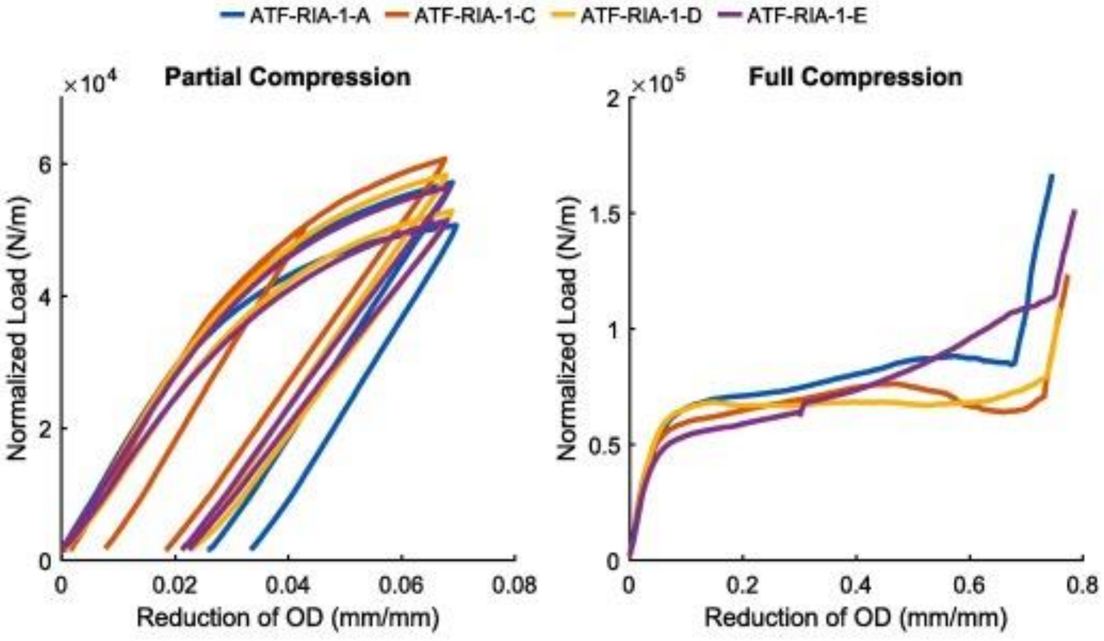


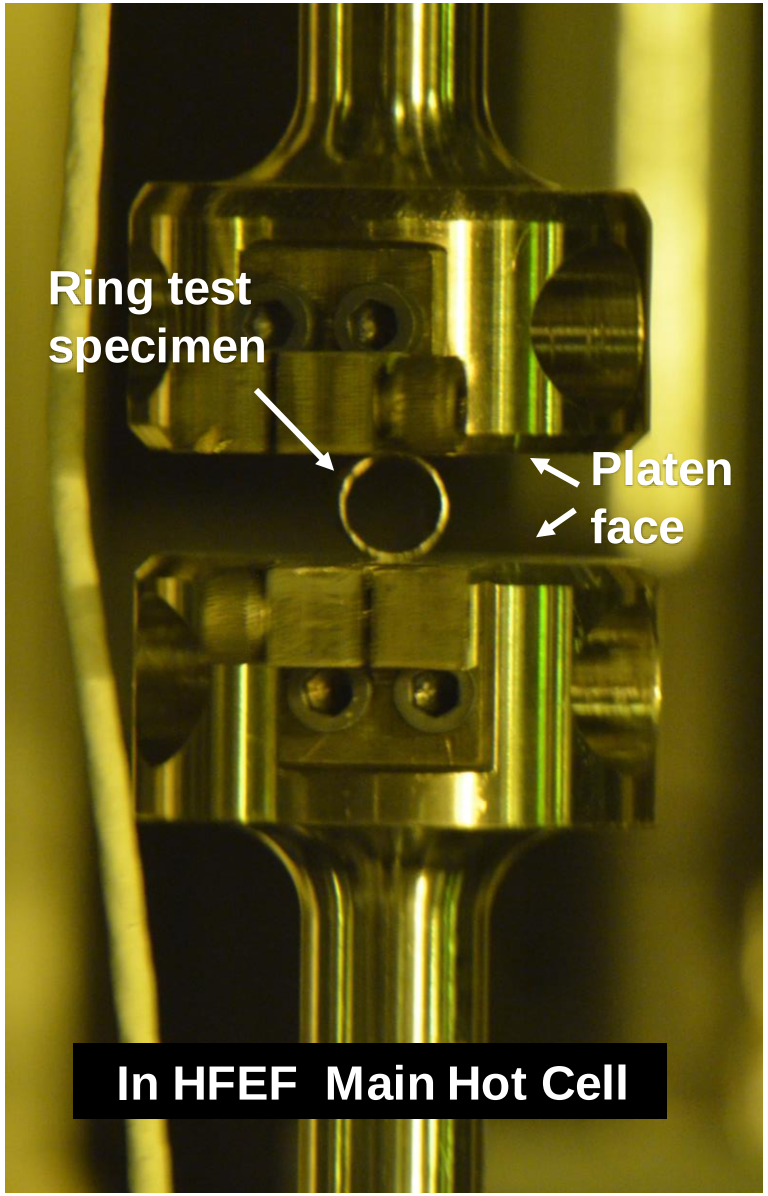
Ring Compression Testing

- Simple compression, using a ring specimen
- Often used for post-transient ductility
- Room and elevated temp
- Example: Post-TREAT testing for ductility

Partial compression for yielding, limited retained ductility

Full compression for cracking, ductility assessment





Folsom et al, *Nuc. Eng. Design*, 2023 https://doi.org/10.1016/j.nucengdes.2023.112509

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