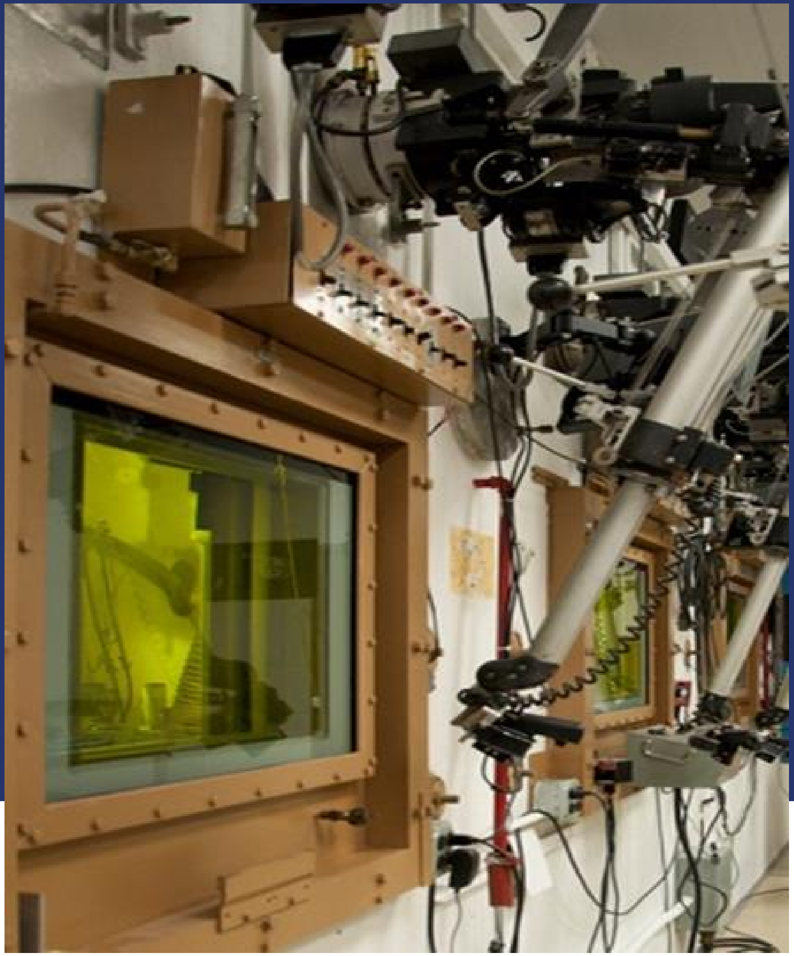
Laboratory Testing and Evaluation of Unirradiated and Neutron Irradiated Additively Manufactured Alloys

Paula Freyer
Fellow Engineer/Metallurgist
Westinghouse Electric Company LLC
Global Technology Office
Churchill Laboratory Services



NRC Public Meeting
Additive Manufacturing for Reactor Materials & Components

North Bethesda, MD
November 28-29, 2017

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Laboratory Testing and Evaluations
– Additively Manufactured (AM) Alloys –

AM 316L

- Completed significant testing and evaluation of unirradiated and 0.8 dpa irradiated samples
- All work performed under WEC* sponsorship
- Samples in storage in Westinghouse Hot Cells
- Additional work on these samples not currently being pursued
- Objective: thimble plugging device insertion into commercial PWR(s) in late 2018

AM Alloy 718

- Completed significant testing and evaluation of unirradiated and 0.8 dpa irradiated samples
- All work performed under WEC* sponsorship
- Samples in storage in Westinghouse Hot Cells
- Aggressively pursuing additional funding (DOE NSUF#) to perform further work

AM Zircaloys

- Samples irradiated to 1, 2 and 3 dpa under WEC* sponsorship
  - 1 dpa irradiations completed
  - 2 dpa irradiations completed 2018
  - 3 dpa irradiations completed 2019
- PIE work will initiate in early 2018 under DOE NSUF# sponsorship
- Aggressively pursuing additional funding to perform further work – likely award in Jan 2018

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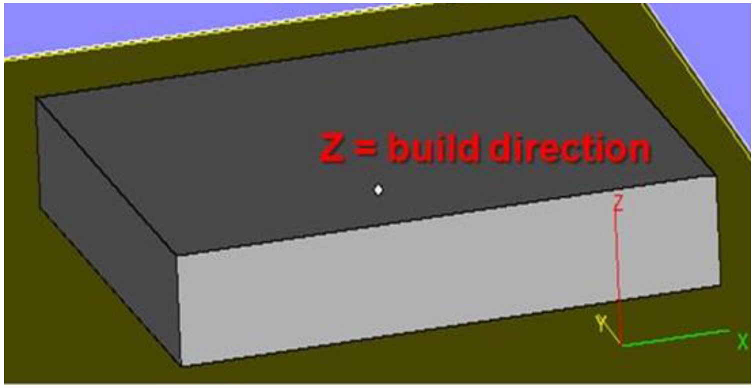
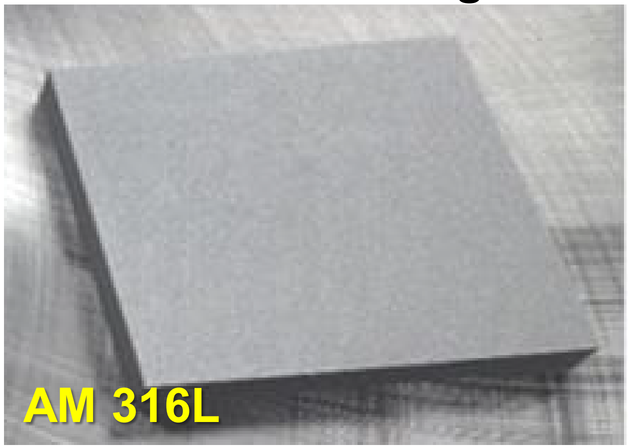
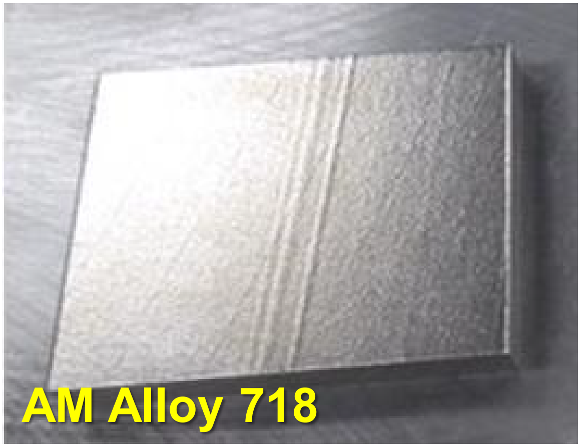
* WEC = Westinghouse Electric Company LLC
# NSUF = Nuclear Science User Facilities

Laboratory Testing and Evaluations
– Typical Approach –

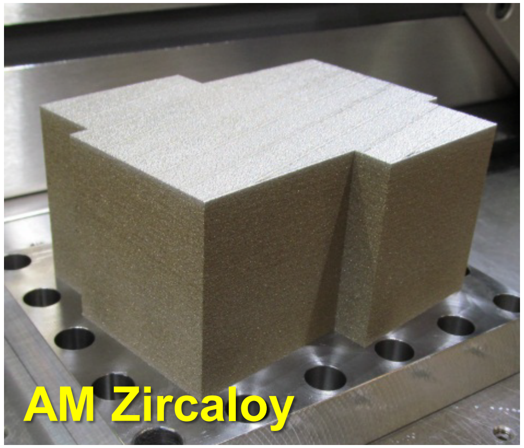
- DMLS block
- Microstructural analysis of as-printed material
- EDM wire cut AM ‘quads’ from X, Y, Z directions and conventional quads from T, L directions
- Heat treat quads
- Neutron irradiate subset of heat treated quads
- Laboratory testing and evaluations of unirradiated, irradiated, AM and conventional materials at Westinghouse







~106 x 106 x 16 mm



~108 x 90 x 56 mm

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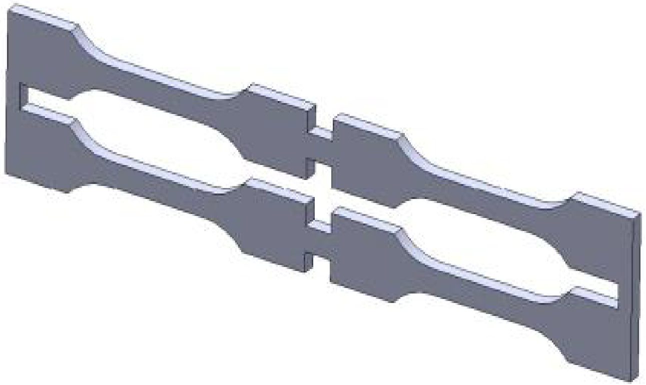
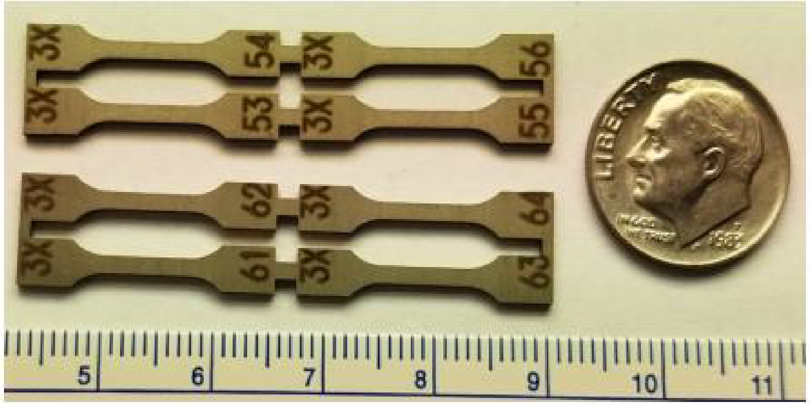
Laboratory Testing and Evaluations
– 'Quad' Miniature Tensile Specimen Geometry –

- Specimens wire EDM cut from test materials as four connected miniature tensiles = 'quads'
- EDM surfaces not polished prior to tensile testing
- Nominal dimensions of individual miniature tensile specimens:
  - L = 23 mm (~ 0.91 inch)
  - W_{gauge} = 1.52 mm (~ 0.06 inch)
  - T = 1 mm (~ 0.04 inch)
- Specimens irradiated in MIT reactor as quads and subsequently separated into individual miniature tensile specimens inside Westinghouse's hot cell





Miniature tensile specimen quads.
Scale in centimeters.

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Scope of Laboratory Testing and Evaluation

- Slight variations for each of the 3 alloys however significant portions of the testing/evaluations are identical
- Includes but not limited to:
  - Radiation measurements
  - Chemistry evaluations (ICP-MS and/or ICP-OES)
  - Immersion density measurements
  - Microhardness
  - Light optical and scanning electron microscopy (unetched and etched)
  - Electron backscattered diffraction (EBSD)
  - Transmission electron microscopy
  - Room and elevated temperature tensile testing with digital image correlations/advanced video extensometry
  - Fractography
  - Hydrogen content analysis
  - Autoclave corrosion testing
  - FIB analysis of surface deposits

Significant materials evaluations completed for AM 316L and AM Alloy 718.

Significant evaluations for AM Zircaloy are funded and will begin Jan 2018.

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Example: AM 316L Testing Program Overview

- Utilized both conventional 316L plate and AM DMLS printed ‘blocks’
  - AM blocks used to reduce/eliminate potential influence of part geometry on material microstructure and tensile properties
- Miniature tensile specimens wire EDM cut from:
  - plate material in transverse (T) and longitudinal (L) directions
  - AM printed block in ‘X’ and ‘Y’ directions (two directions in build plane)
- Miniature tensile specimens irradiated in MIT reactor for a ~5 months to a damage dose of ~0.8 dpa
- Analysis included: tensile testing, chemical analysis, corrosion testing, focused ion beam cross sectional analysis of surface deposits, light optical microscopy, scanning electron microscopy, fractography, and hardness testing, etc.
  - We have published some microstructural results and a majority of the tensile results

Tensile:
P.D. Freyer, W.T. Cleary, E.M. Ruminski, C.J. Long, P. Xu, *“Hot Cell Tensile Testing of Neutron Irradiated Additively Manufactured Type 316L Stainless Steel,”* 18th International Conference on Environmental Degradation of Materials in Nuclear Power Systems – Water Reactors, Aug 2017, Portland, Oregon.

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AM 316L Testing Summary

- **Tensile testing performed at both room and elevated temperature**
- **12 different tensile test conditions evaluated (next table)**
- **Conventional plate material**
  - **Standard annealed condition (i.e., 1038°C (1900°F))**
  - **ASTM A479/A479M – 17**
  - **ASTM A240/A240M – 16a**
  - **Certified material test report (CMTR) - compliant with all applicable ASTM chemistry and mechanical property requirements**
- **AM material**
  - **Produced as block using DMLS process and 316L (UNS S31673) powder**
  - **Mean build layer thickness of 20 μm (~0.8 mil)**
  - **Standard anneal performed on quads cut from block**

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AM 316L Testing Summary

Summary of 5 material conditions evaluated, including 10 material orientations, and tensile results presented

Number	Irradiation Condition	Conventional or AM	Condition	Orientation Evaluated	Tensile Results Summarized Herein
1	Unirradiated	Conventional Plate	Annealed	L and T	✓
2		AM	Printed (microstructural characterization only)	X and Y	Some microstructural results provided
3		AM	Printed + annealed	X and Y	✓
4		AM	Printed + annealed + long term thermally exposed	X and Y	
5	Irradiated	AM	Printed + annealed + irradiated	X and Y	✓

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AM 316L Testing Summary

Summary of 12 tensile test conditions evaluated

Data Set	Number	Material Condition Description
A	1	L Conventional Unirradiated Room Temperature
	2	T Conventional Unirradiated Room Temperature
B	3	L Conventional Unirradiated Elevated Temperature
	4	T Conventional Unirradiated Elevated Temperature
C	5	X AM Unirradiated Room Temperature
	6	Y AM Unirradiated Room Temperature
D	7	X AM Unirradiated Elevated Temperature
	8	Y AM Unirradiated Elevated Temperature
E	9	X AM Irradiated Room Temperature
	10	Y AM Irradiated Room Temperature
F	11	X AM Irradiated Elevated Temperature
	12	Y AM Irradiated Elevated Temperature

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Conventional Plate and AM Powder Compositions

CMTR reported chemical composition (wt%) for conventional plate material and for powder utilized for the DMLS printed block

Element	316L Conventional Plate UNS S31600/31603 (from CMTR)	316L AM Powder UNS S31673 (from powder supplier)
Fe	Balance	Balance
Cr	16.63	17.00-19.00
Ni	10.03	13.00-15.00
Mo	2.01	2.25-3.00
Mn	1.47	2.00 max
Si	0.23	0.75 max
P	0.04	0.025 max
Cu	0.51	0.50 max
S	0.001	0.010 max
N	0.04	0.10 max
C	0.016	0.030 max
Co	0.32	…

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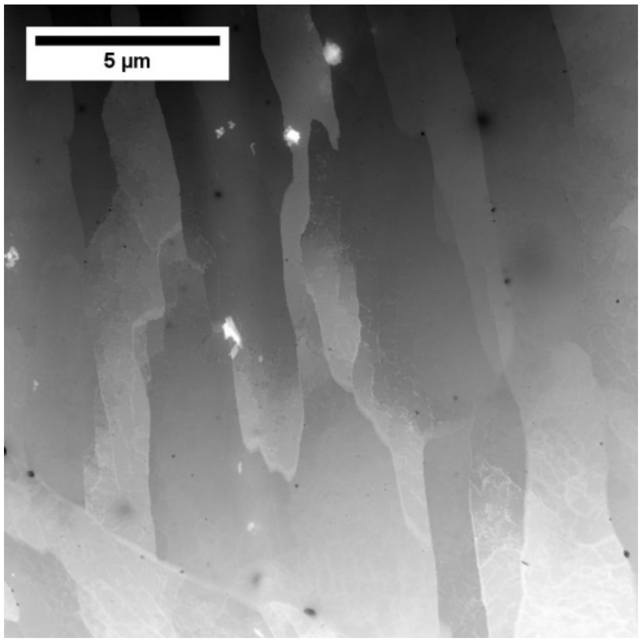
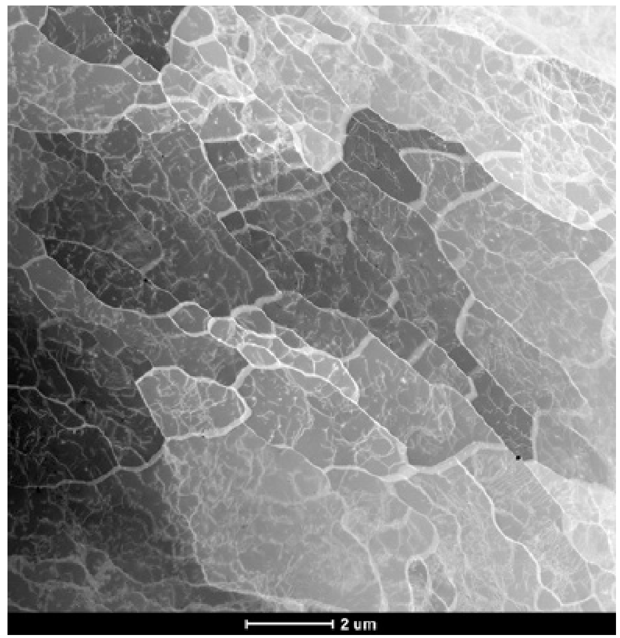
AM 316L Test Material
– As-Deposited Microstructure –



~4,800X



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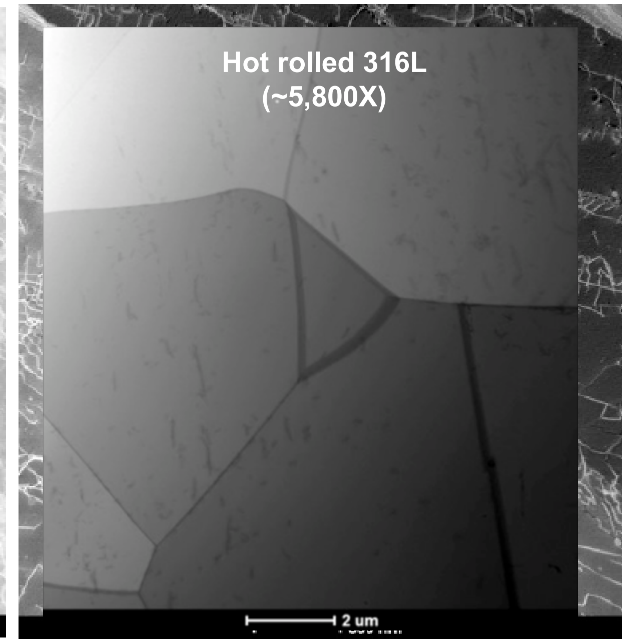

~22,000X

HAADF STEM of columnar grains containing subgrains

ADF STEM of dislocation networks within grains

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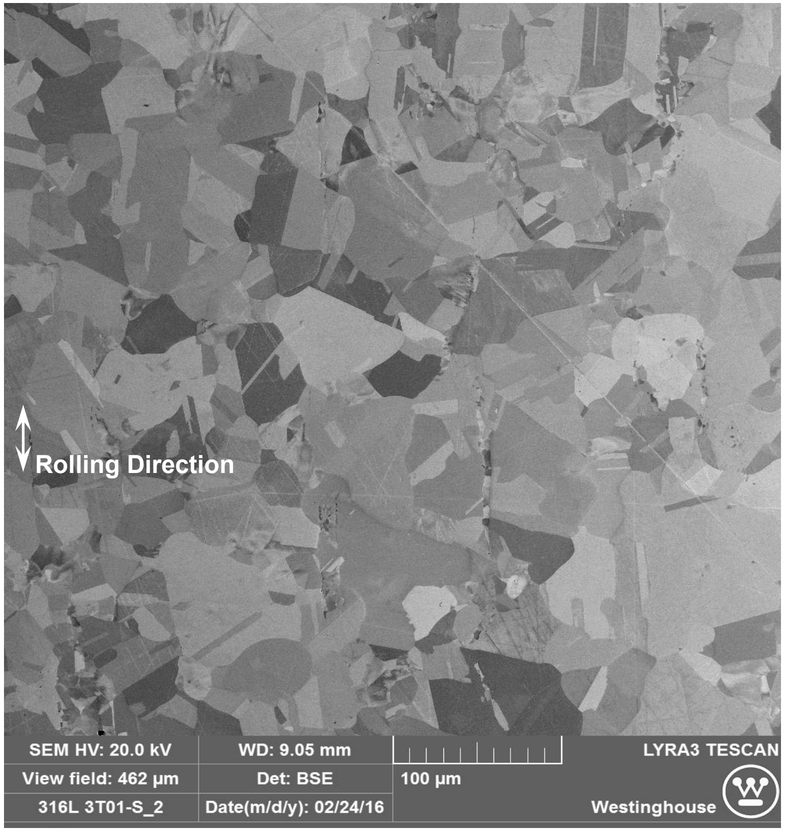
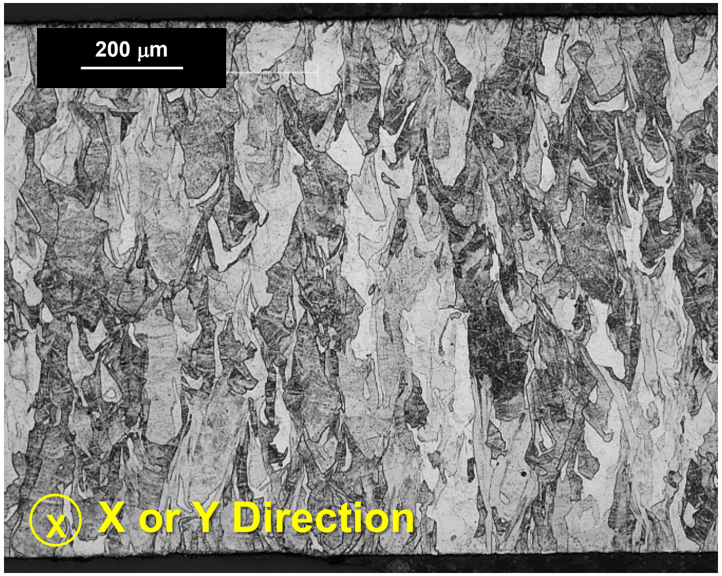
J.J.H. Lim, A.R.C. Malheiros, G. Bertali, C.J. Long, P.D. Freyer and M.G. Burke, "*Comparison of Additive Manufactured and Conventional 316L Stainless Steels*," Microscopy & Microanalysis, suppl. S3; Cambridge 21, Aug 2015, pp. 467-468.

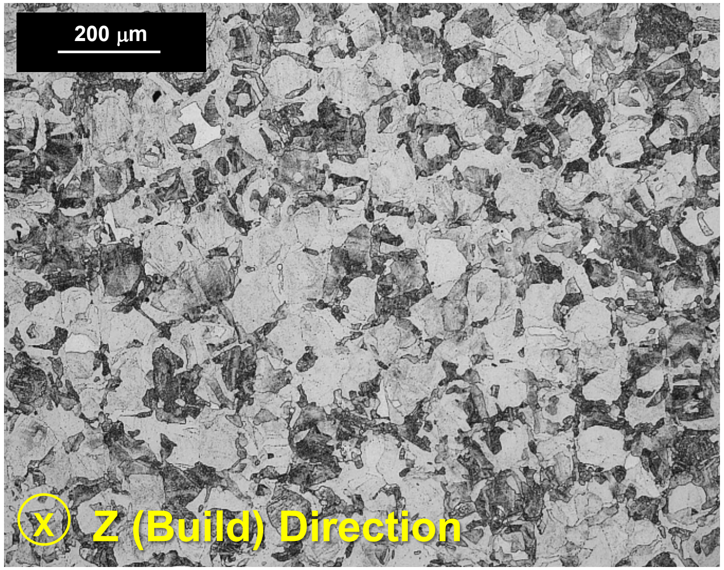
Conventional and AM 316L Test Material
– Heat Treated Microstructures –



Conventional 316L Plate
Backscattered Secondary Electron SEM Micrograph (~220X)



AM 316L
Light Optical Micrographs (~65X)



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Autoclave Corrosion Testing of Conventional and AM 316L

- 30 days flowing autoclave at simulated PWR primary T, P, and chemistry conditions (per EPRI guidelines)
- Morphology and thickness of resulting oxide characterized using FIB and SEM
- Oxide thickness → estimate corrosion rate

Similar corrosion rates of conventional and AM alloys - base material manufacturing method did not influence corrosion rate.

More in-depth corrosion testing is needed.

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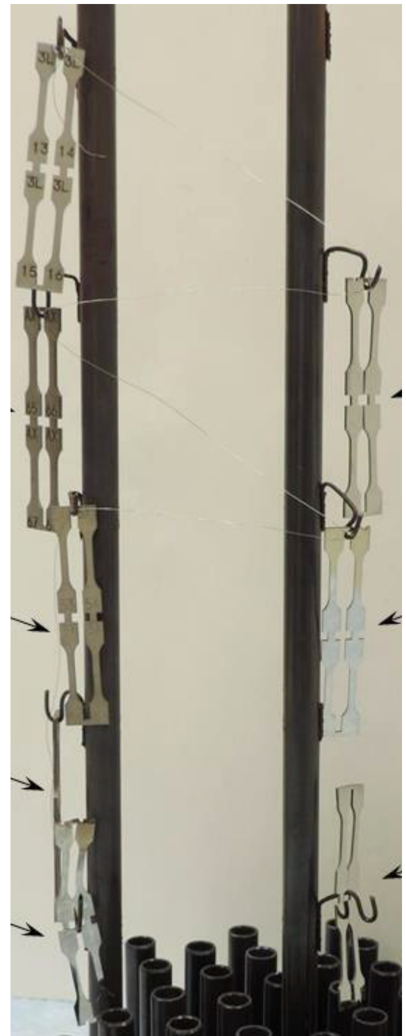
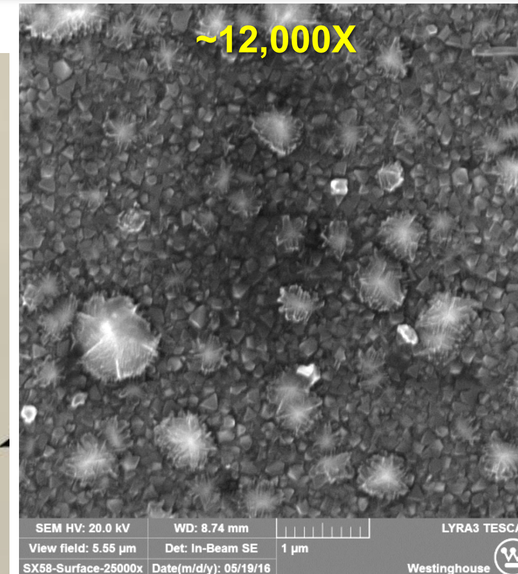
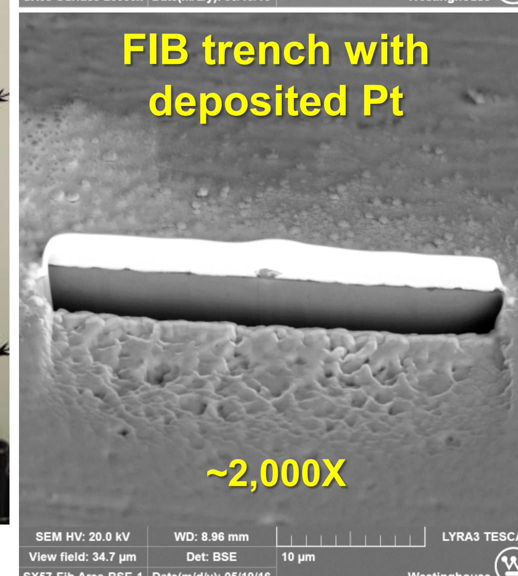
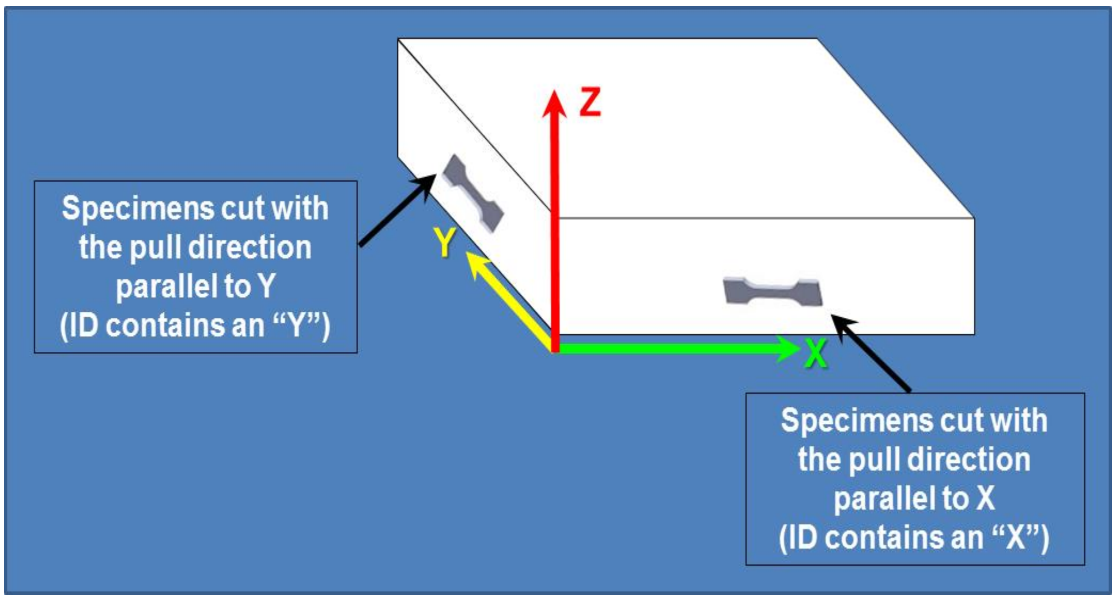


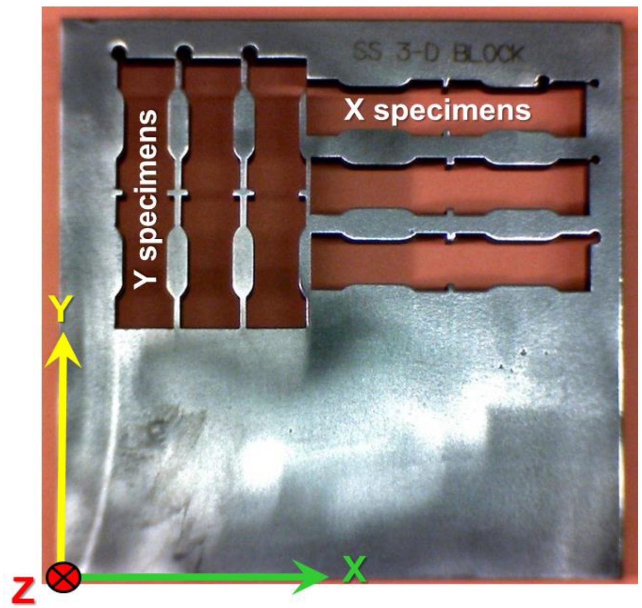




Tensile Specimen Orientations

- X and Y orientations cut from AM block
- No Z tensile specimens (AM block thickness not sufficient to allow for specimens in this orientation)
- For conventional plate material, L and T directions same as typically used to describe plate product orientations relative to rolling direction





Wire EDM cutting of quads from AM 316L test block

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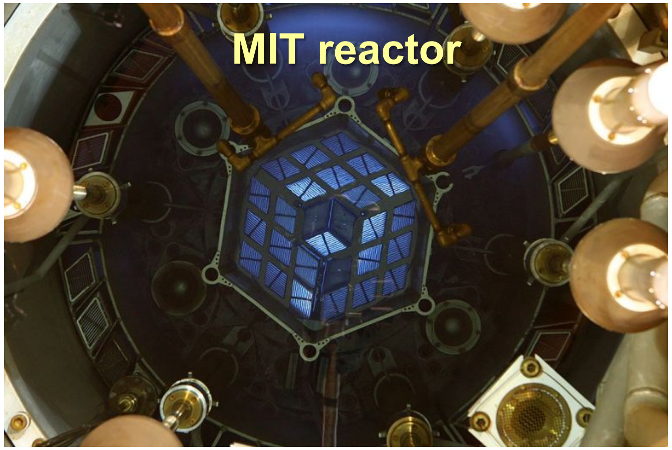
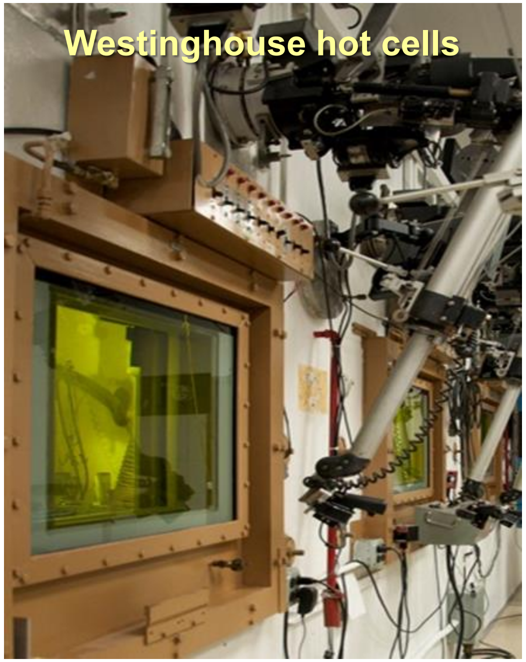
Irradiated Material Description

- 2015 irradiation of AM quads in MIT reactor for ~5 months to fluence:
  - 0.8 x 10^{21} n/cm^2 thermal
  - 1.2 x 10^{21} n/cm^2 (E > 0.1 MeV)
  - 6.5 x 10^{20} n/cm^2 (E > 1.0 MeV)
- Damage dose of ~ 0.8 dpa
- Irradiated close to core center (peak flux)
- Irradiated ~298°C (568°F)
- Quads cooled at MIT for ~5 months prior to shipment to Westinghouse Hot Cells
- Total of 6 AM quads irradiated, 3 were AM 316L quads (12 miniature tensiles)





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Radiation Measurements of Irradiated Quads

- Measurements for three irradiated AM 316L quads
- Near contact dose rates of ~150 R/hr
  - all work performed inside Westinghouse hot cells

Quad Identification Numbers	Measured and Calculated Dose Rates			
	At ~1 m (39")	At ~0.3 m (12")	At ~2.5 cm (1")	At ~1.3 cm (0.5")
	Measured Value	Measured Value	Calculated Value	Calculated Value
	mR/hr		R/hr	
SX01-SX04	36	260	37	150
SX49-SX52	35	230	33	132
SY25-SY28	38	250	36	144

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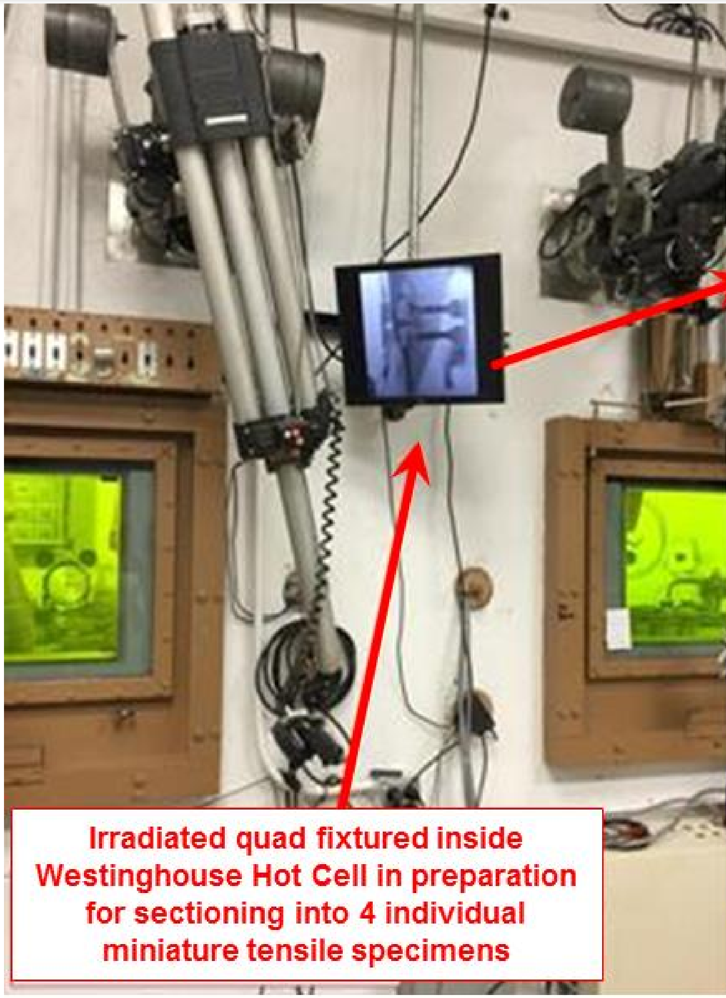
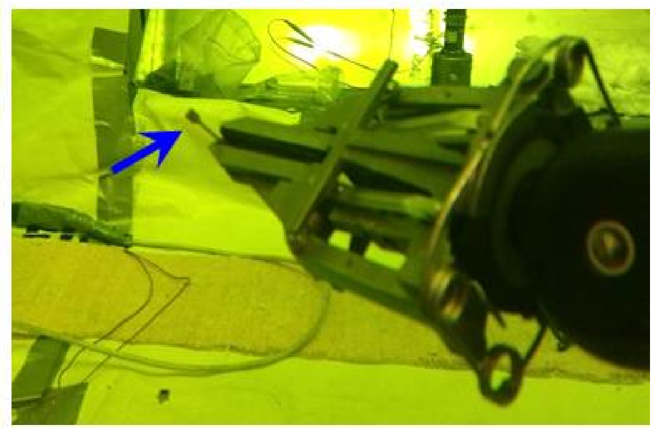
In-Cell Sectioning of Irradiated Quads



Irradiated quad fixtured inside Westinghouse Hot Cell in preparation for sectioning into 4 individual miniature tensile specimens

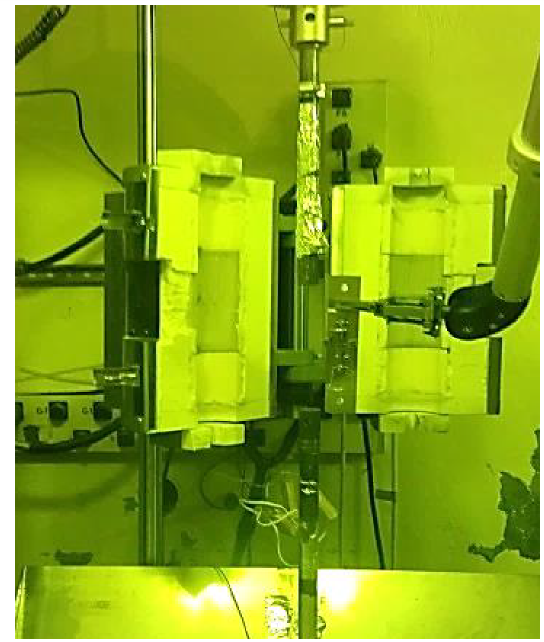
Close up view of irradiated quad shown on video monitor



Individual miniature tensile specimen (blue arrow) held by manipulator

Tensile Testing Approach

- Instron screw driven tensile machine with:
  - Instron Digital Image Correlation/Advanced Video Extensometer (DIC/AVE)
  - Instron 5 kN load cell
- Custom designed and fabricated specimen holding fixture
  - optimized to specifically be used with hot cell manipulators
- Specimens first loaded into fixture and then fixture installed onto pull rods of in-cell tensile machine
- DIC not utilized for elevated temperature tests



Alignment of pin holes on specimen holding fixture with pin holes on tensile machine pull rod clevises inside Low Level Hot Cell

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In-Cell DIC/AVE

- First must speckle contrast mark specimens
- DIC camera captures images during test
- DIC software follows movement of speckle points located within gauge length
- Images collected during testing and processing of strain data occurs after test
- DIC and load cell calibrated in accordance with ASTM specifications

DIC system inside Low Level Hot Cell (marked with yellow arrow)

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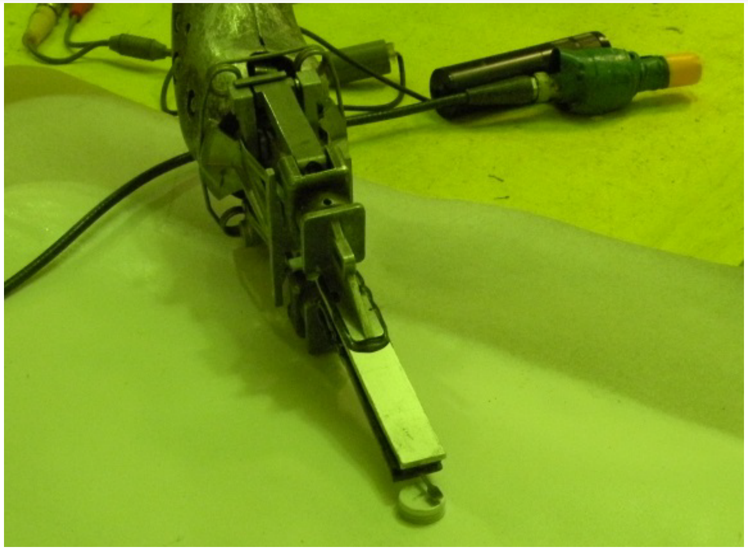
Speckle Marking for DIC

- **Optimum approach developed for speckle marking**
- **Numerous different paints and application techniques initially evaluated**
- **Optimum: spray white paint ~0.3-0.6 m (1-2 feet) above specimen and allow paint mist to settle down onto specimen surface**
- **Repeatedly produced miniature tensile specimens with excellent speckle patterns**



Placement of individual irradiated miniature tensile specimen onto small raised platform and example of good speckle pattern

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Tensile Test Results

Reference Document or Data Set	UTS, MPa	0.2% YS, MPa	EL, %	RA, %
Room Temperature ASTM Specification Minimums and CMTR Values				
Conventional Unirradiated ASTM Spec A479	485	170	30	40
Conventional Unirradiated ASTM Spec A240	485	170	40	Not specified
Conventional Unirradiated CMTR for 316L	577	260	57	74
AM Unirradiated ASTM Spec F3184-16	515	205	30	40
Room Temperature Test Results				
Data Set A: Conventional Unirradiated	618	282	63	85
Data Set C: AM Unirradiated	605	357	48	77
Data Set E: AM Irradiated	652	427	43	75
Elevated Temperature Test Results				
Data Set B: Conventional Unirradiated	452			
Data Set D: AM Unirradiated	450			
Data Set F: AM Irradiated	493			

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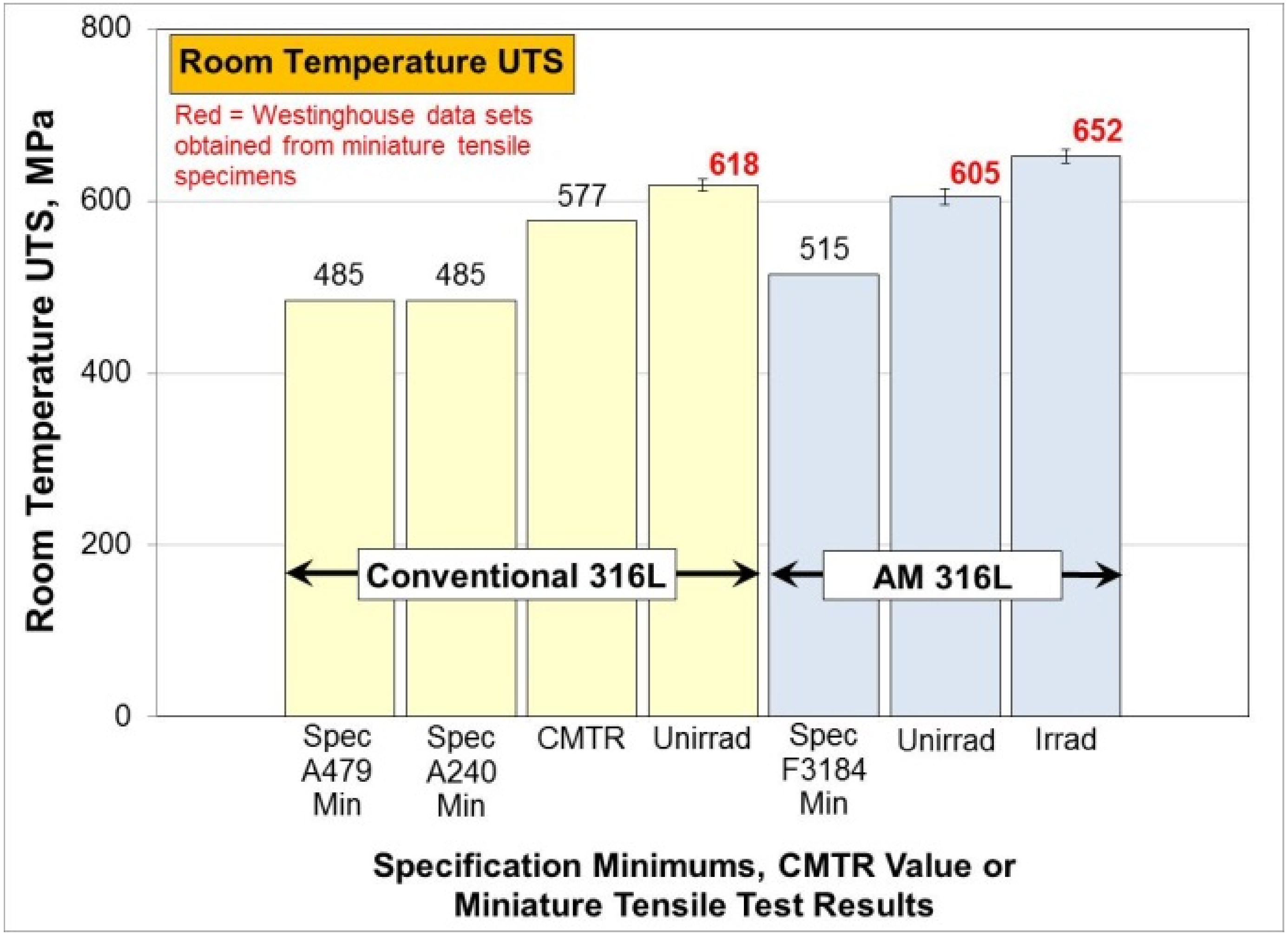
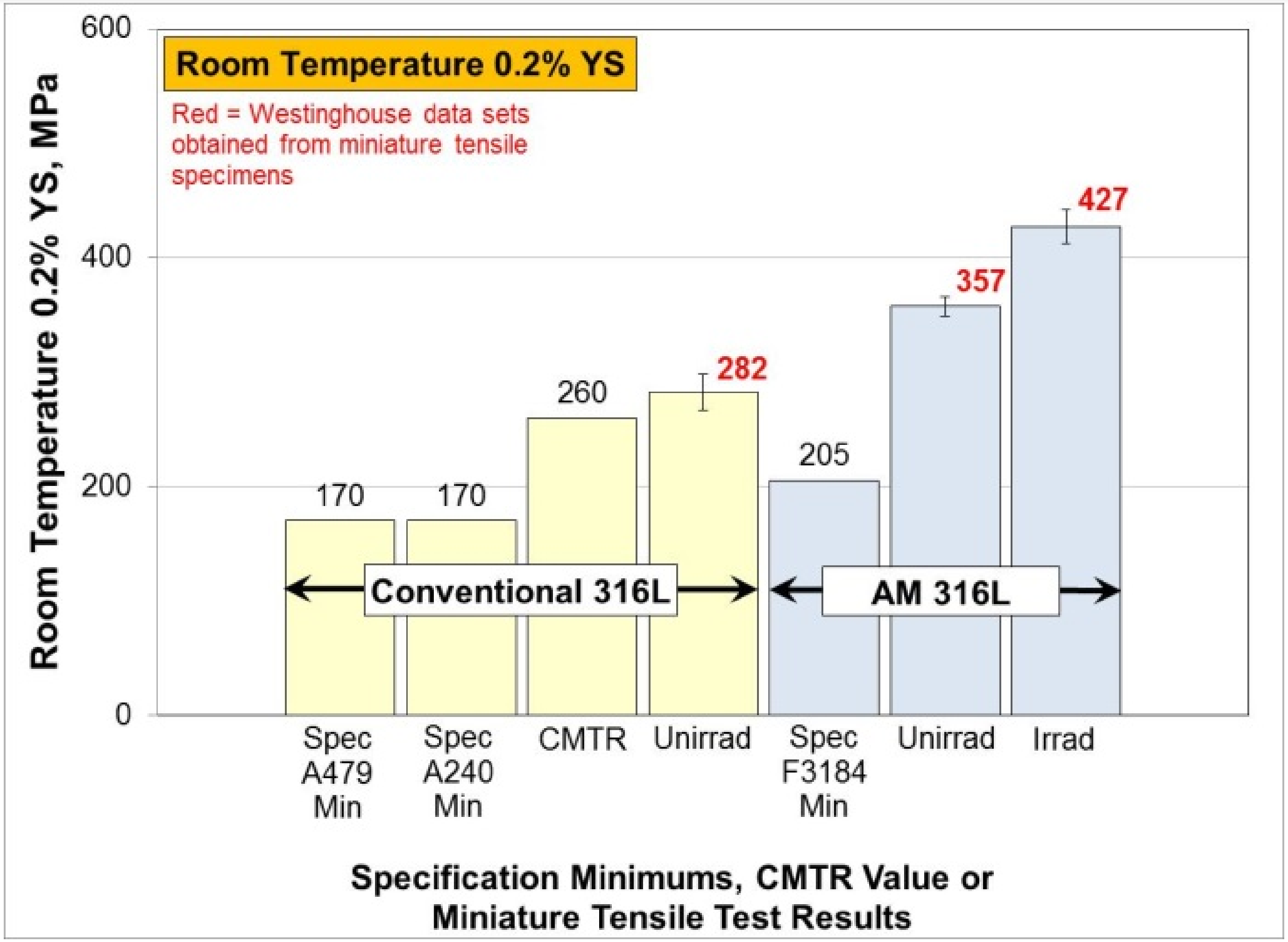
Tensile Test Results - UTS



Tensile Test Results – 0.2% YS



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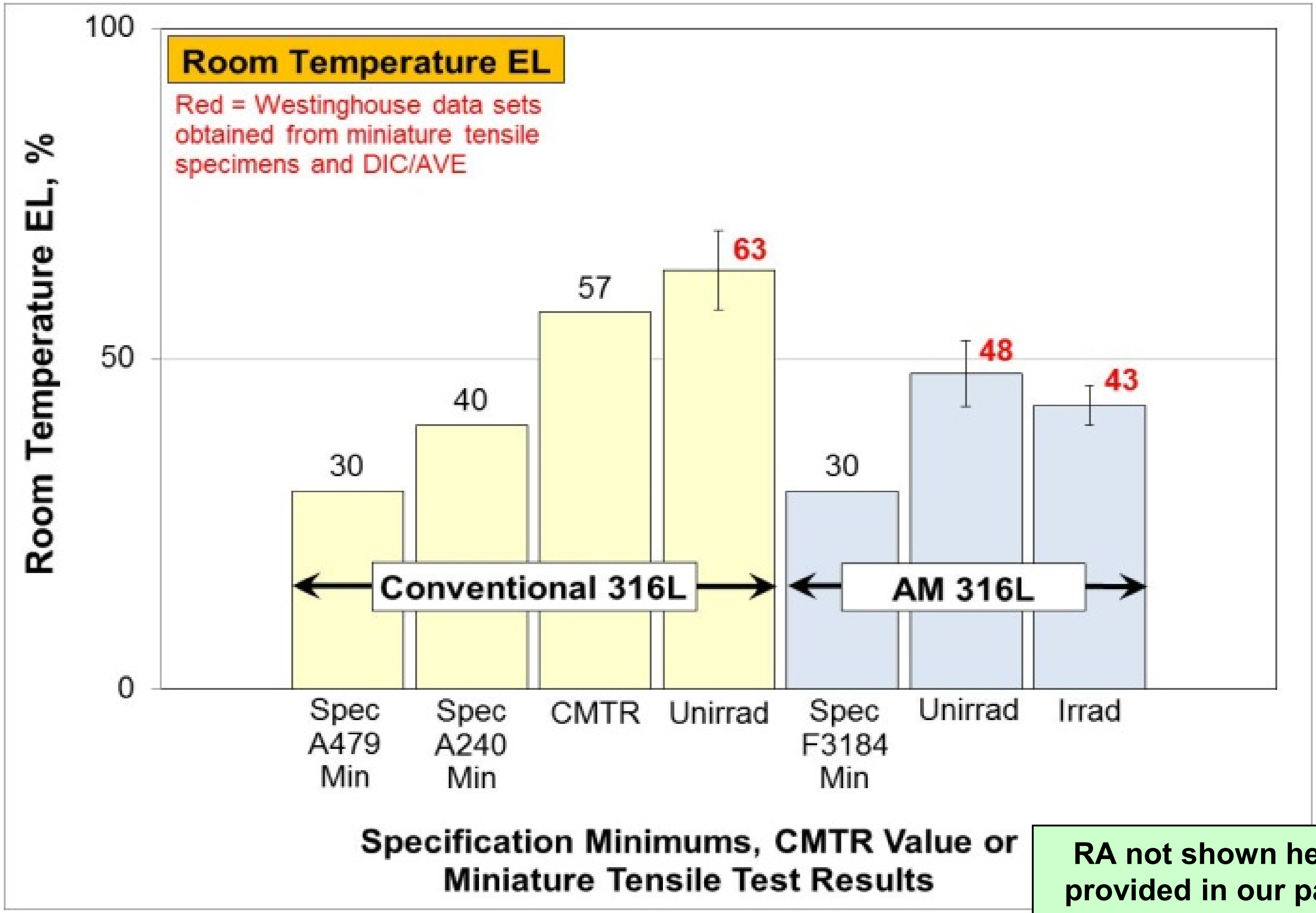
Tensile Test Results - %EL



RA not shown here but is provided in our paper, AM values are in the range of 75-77%
(measured via SEM fractographic images)

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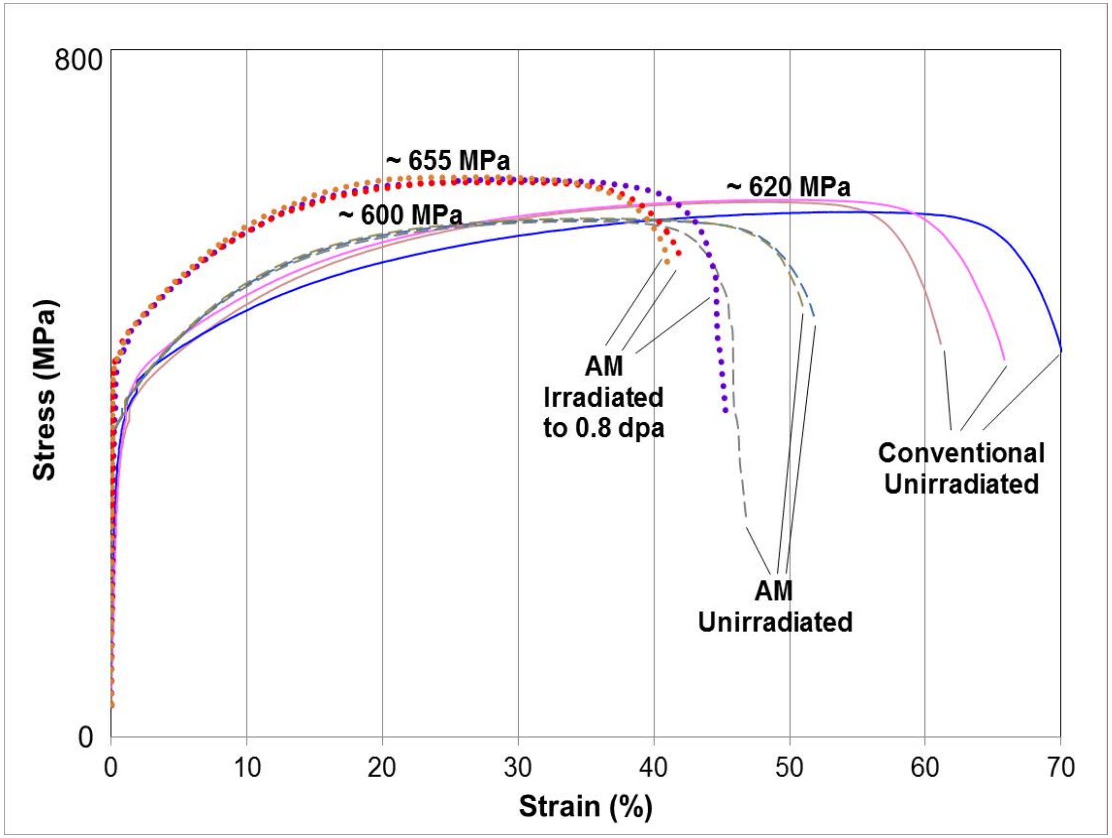
Examples of Stress Strain Curves

- Good reproducibility
- Unirradiated conventional
  - highest strain to failure of ~60-70%
  - maximum stress of ~620 MPa
- Unirradiated AM 316L
  - lower strain at fracture values of ~48-52%
  - slightly lower maximum stress of ~600 MPa
- Irradiated AM 316L
  - further decrease in strain to ~40-45%
  - increase in maximum stress to ~655 MPa



Stress strain curves for nine miniature specimens tested at room temperature

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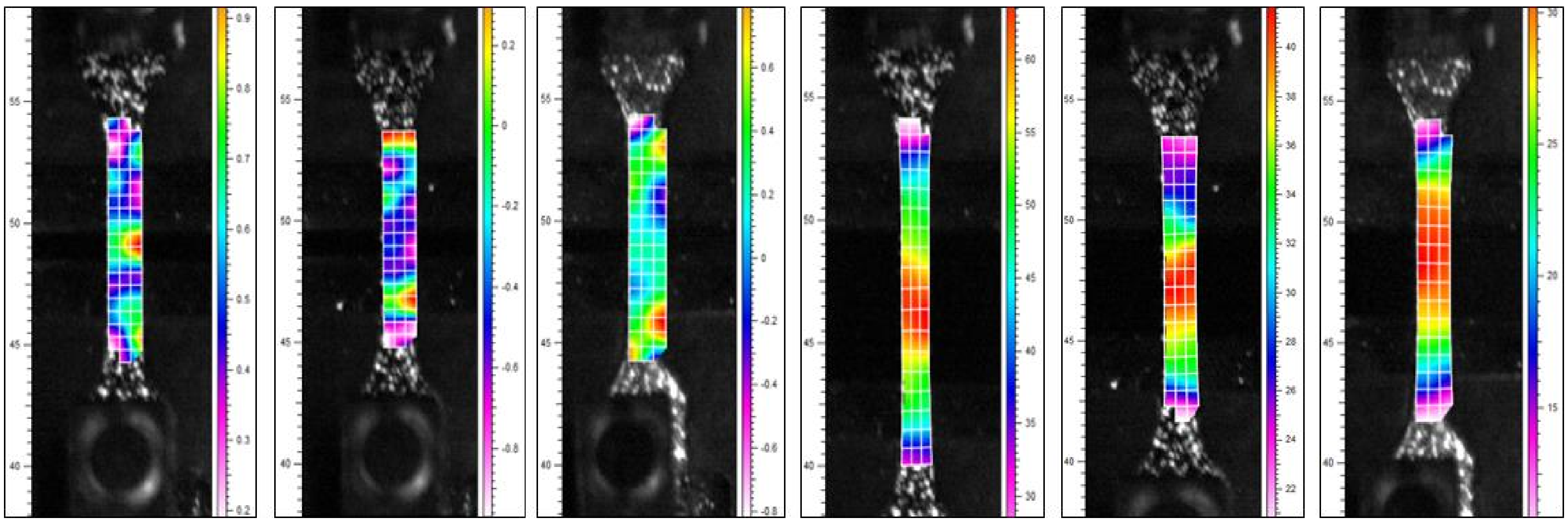
Examples of DIC Axial Strain Distribution Maps

- Maps at 345 MPa (50 ksi) and UTS
- Note speckled grip ends can be seen in most images
- Maps at same dimensional scale but not same strain scale

Sample 1 | Sample 2 | Sample 3 | Sample 1 | Sample 2 | Sample 3



Unirradiated Conventional	Unirradiated AM	Irradiated AM	Unirradiated Conventional	Unirradiated AM	Irradiated AM
			605 MPa EL = 68.3%	599 MPa EL = 47.6%	649 MPa EL = 45.3%

← At 345 MPa → ← At UTS →

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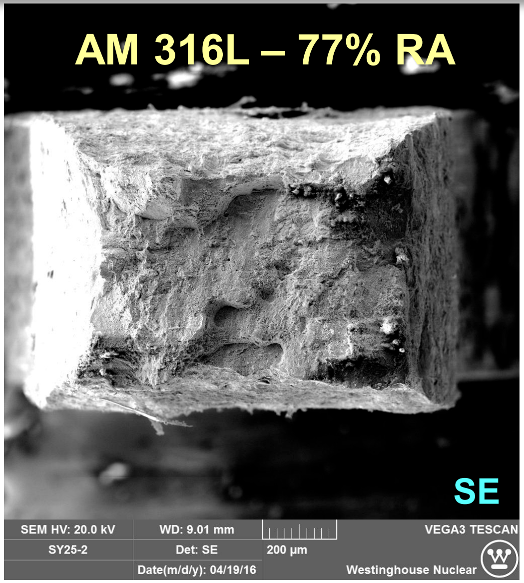
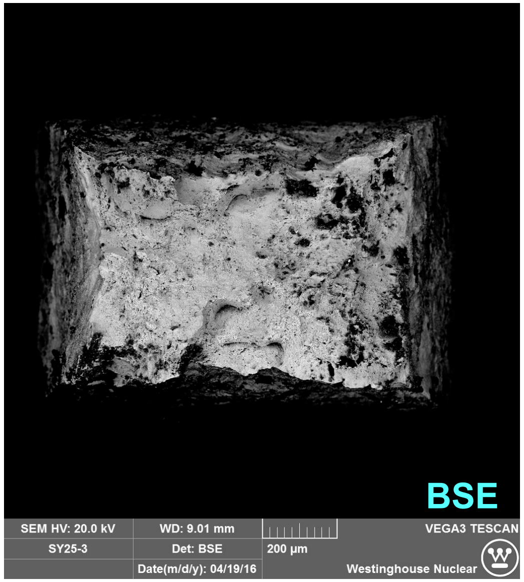
Summary and Conclusions - 1

General Observations

- Highly activated miniature tensile specimens successfully tested in-cell utilizing custom designed and fabricated specimen holder and DIC/AVE
- Total of 46 conventional and AM 316L specimens tested at both room temperature and 300°C (572°F)
- Results obtained are encouraging - work continues towards development of AM technologies for fuel-related components
  - – including testing of higher damage dose materials in 2017-2019
- Significant near term goal: fabrication and delivery of lead test component to Westinghouse nuclear utility customer for in-reactor insertion
- Tensile test data from Z direction is needed
- Data sets show relatively low standard deviations





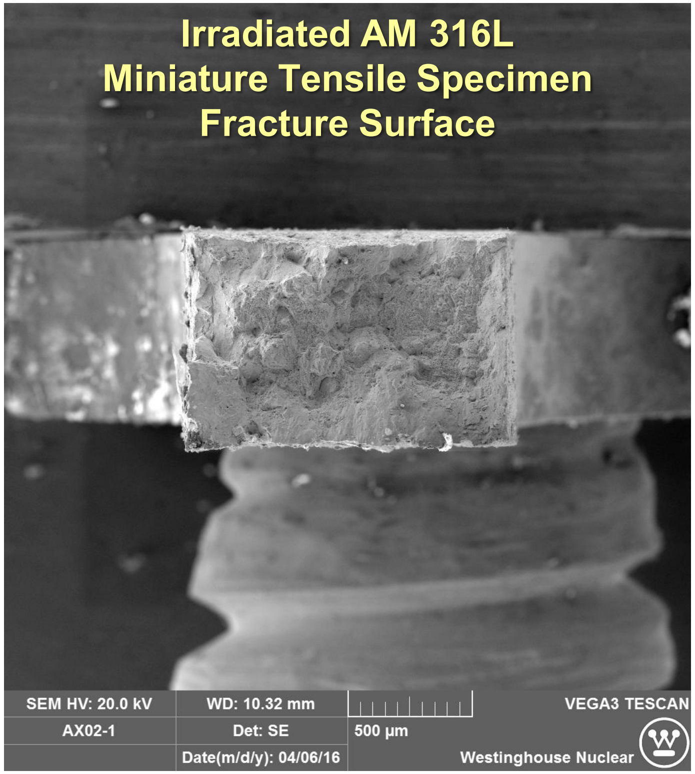
Summary and Conclusions - 2

Room Temperature Tensile Results

- Unirradiated and irradiated AM 316L tensile properties exceed ASTM AM 316L specifications, and generally significantly exceed minimum property requirements
- Unirradiated AM 316L (compared to conventional 316L)
  - UTS value nearly identical
  - YS higher by approximately 75 MPa
  - EL and RA lower by ~8-15%
- Irradiated AM 316L (compared to unirradiated AM 316L)
  - UTS and YS higher by ~50 MPa and 70 MPa, respectively
  - EL and RA lower by ~2-5%

Elevated Temperature Tensile Results

- Unirradiated AM 316L UTS essentially identical to conventional 316L
- Irradiated AM 316L UTS higher than unirradiated AM 316L by ~45 MPa



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Acknowledgements

- Westinghouse Nuclear Fuels, and specifically Mr. Zeses Karoutas, Chief Engineer, for his unwavering support of this work
- Westinghouse Supply Chain Management for their outstanding assistance
- Mr. Gordon Kohse of MIT for exceptional assistance regarding irradiation of test specimens
- Mr. Jason Boyle of Westinghouse Hot Cell Facility for excellent work performing the tensile tests
- Westinghouse Hot Cell technicians and Radiation Safety Officer for outstanding laboratory evaluations and radiological support

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