U.S. DEPARTMENT OF ENERGY | Nuclear Energy

Nuclear Science User Facilities

Industry Program update 2016

(INL/MIS-14-32147) Rev 4

NSUF Nuclear Science User Facilities

John H. Jackson
Industry Program Lead

FY 2016 NSUF Annual Program Review
Germantown, MD
November 1-3, 2016

Annual NSUF Industry Advisory Committee meeting

- **~Annual meeting hosted by Electric Power Research Institute (EPRI)**
  - Trending toward 18 month cycle
- **Representation from EPRI, NRC, Areva NP, Westinghouse, GE, Rolls Royce, EdF, Exelon, Duke, MAI, NEI, Dominion (others are welcome… always expanding)**
- **Opportunity for industrial participation and guidance**
  - Prioritize industry research needs for ATR and PIE capability
  - Maintain NSUF as an asset for industry
  - Build working relationships between NSUF staff and industry
- **Most recent meeting June 30-July 1, 2015**
- **Next ANIAC ~February-March 2017 (coincident with EPRI NPC?)**

2016 base activities

- **Replacement of HFEF Instron furnace**
  - Current furnace is single zone and lacks proper insulation
  - New, 3 zone furnace with custom insulation inserts at top and bottom
- **Addition of extensometer to HFEF Instron**
  - Currently rely on crosshead displacement and compliance measurement
  - Require measurement across specimen gage length
- **Implementation of SCC software**
  - Current software is DOS based and has single point of failure
  - Replace with Labview version
- **CRADA support**
  - X-750 further investigation
- **Loop 2A flow restriction mitigation**

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Current Industry Programs

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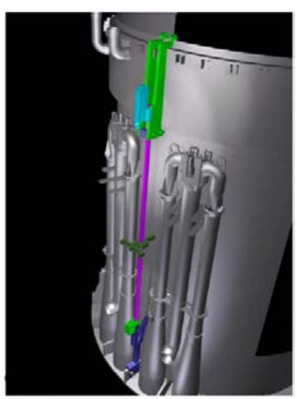
- **EPRI Pilot Project: Irradiation and PIE of alloys X-750 and XM-19 (three phases)**
  - Irradiate to three fluences (5 X 10^{19}, 2 X 10^{20}, 1 X 10^{21} n/cm^2) at ATR
  - Perform tensile, CGR, FT tests, and TEM microstructural analyses
- **EPRI Pilot Project: Zirconium alloy irradiation growth**
  - Measure hydrogen assisted irradiation induced growth under BWR relevant conditions.
  - Characterize irradiation induced defects and correlate to growth
- **NRC: Irradiation and testing of austenitic SS**
  - Sensitized 304 and 304L weld HAZ
  - Piggyback with EPRI specimens
- **GE-Hitachi: Irradiation Testing of LWR Additively Manufactured Materials (new in 2017)**
  - Direct Metal Laser Melted (DMLM) 316 SS and 718
  - Irradiation at ATR to 0.5-1.0 dpa plus IASCC, FT, and TEM

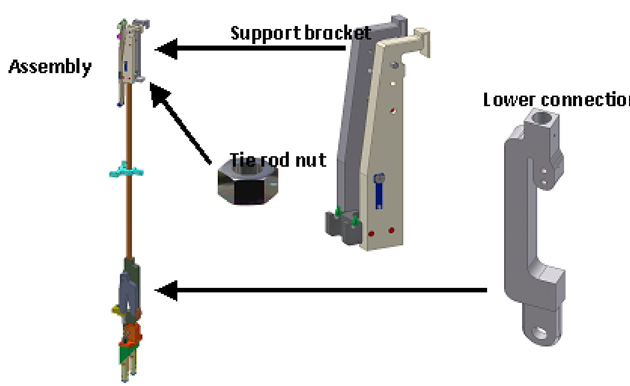
Completed Industry programs (2016)

- **CNL: TEM and micro-hardness of Candu reactor springs**
  - Alloy X-750 garter springs
  - FIB and TEM of highly irradiated spring sections (between 5 and 55 dpa)
- **EPRI: Conductivity measurements in irradiated Zr**
  - Utilize material from current pilot project
  - Correlate conductivity with radiation damage
  - Strategic Partnership Project (formerly WFO)

EPRI Pilot Project 1: Irradiation and PIE of alloys X-750 and XM-19

- **Alloys X-750 and XM-19 are used in many structural applications in BWRs ranging from original equipment to modifications and repair hardware**
  - SCC and fracture toughness data in BWR water chemistry conditions are rather limited, particularly when exposed to neutron irradiation
- **A multi-year program is in place to examine the SCC and fracture toughness behavior of these materials under a variety of BWR conditions, both un-irradiated and irradiated**





EPRI Pilot Project 1: Irradiation and PIE of alloys X-750 and XM-19

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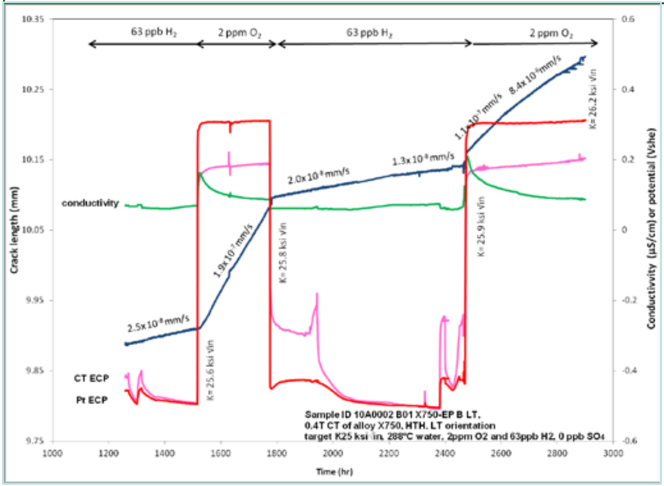
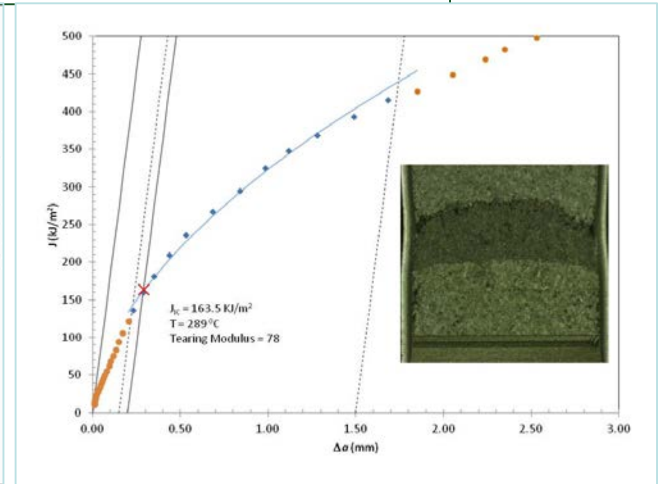
- Irradiate X-750 and XM-19 in ATR Center Flux Trap with new Loop 2A
- Measure crack growth rate and fracture toughness in simulated BWR conditions using new IASCC test systems

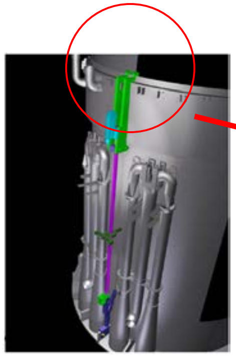


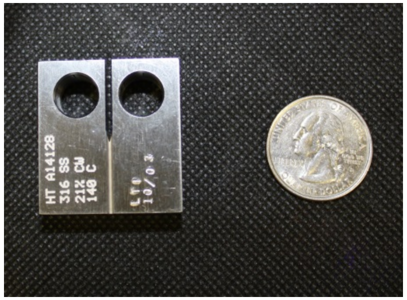
SCC of un-irradiated inconel X-750



Fracture toughness of un-irradiated X-750

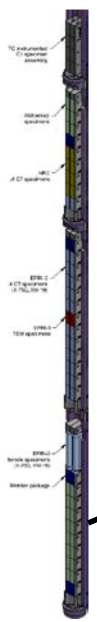
IASCC test systems



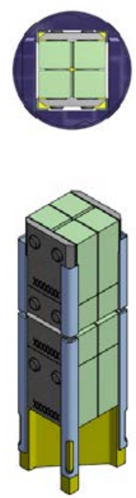


EPRI-3 irradiation completed

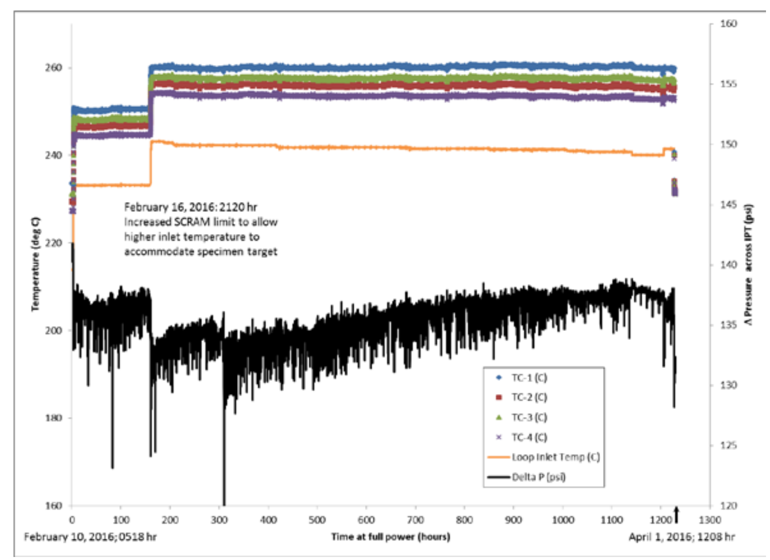
- **Target fluence = 1.0 X 10^{21} n/cm^2 (E>1 MeV)**
- **Two cycles to reach target**
  - Cycle 155 B (50 days) completed April 12, 2014
  - Cycle 158 B (51 days) completed April 1, 2016
    - Delayed due to coolant flow restriction in experiment and necessary mitigation
- **Modified experiment between cycles to correct flow restriction**



Capsule holder “A” replaced with hydraulic and thermal equivalent



Alignment system to prevent package “twisting”



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Completed EPRI-3 and EPRI-1 as-run analyses

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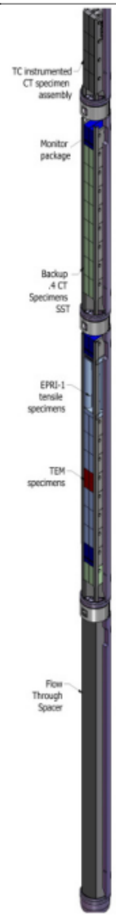
EPRI-1: target fluence 5.0 X 10^{19} n/cm^2



EPRI-1 Holder	Package	Calculated Flux (n/cm^2-sec)	Calculated Fluence (n/cm^2)
EPRI-1D	EPRI-1D4	2.02E+13	9.42E+18
	EPRI-1D3	2.75E+13	1.28E+19
	EPRI-1D2	3.49E+13	1.63E+19
	EPRI-1D1	4.18E+13	1.95E+19
EPRI-1C	EPRI-1C10	6.31E+13	2.95E+19
	EPRI-1C9	7.13E+13	3.33E+19
	EPRI-1C8	8.01E+13	3.74E+19
	EPRI-1C7	8.63E+13	4.03E+19
	EPRI-1C6	9.25E+13	4.31E+19
	EPRI-1C5	9.48E+13	4.42E+19
	EPRI-1C4	9.91E+13	4.62E+19
	EPRI-1C3	1.04E+14	4.84E+19
	EPRI-1C2	1.05E+14	4.91E+19
	EPRI-1C1	1.07E+14	5.00E+19
EPRI-1B	EPRI-1B14	1.09E+14	5.09E+19
	EPRI-1B11-13	1.12E+14	5.23E+19
	EPRI-1B10	1.15E+14	5.35E+19
	EPRI-1B9	1.14E+14	5.33E+19
	EPRI-1B8	1.15E+14	5.35E+19
	EPRI-1B7	1.15E+14	5.35E+19
	EPRI-1B6	1.14E+14	5.33E+19
	EPRI-1B5	1.15E+14	5.35E+19
	EPRI-1B4	1.14E+14	5.33E+19
	EPRI-1B3	1.13E+14	5.29E+19
	EPRI-1B2	1.14E+14	5.33E+19
	EPRI-1B1	--	--
EPRI-1A	Spacer	--	--
	Spacer	--	--
	Spacer	--	--
	Spacer	--	--
	Spacer	--	--
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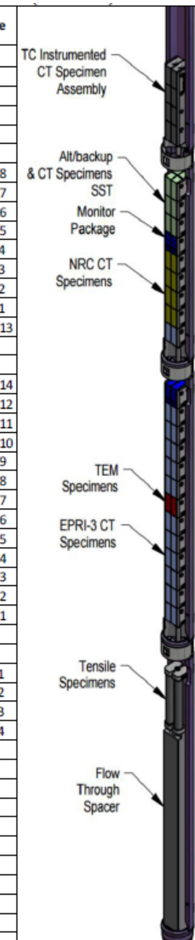
EPRI-3: target fluence 1.0 X 10^{21} n/cm^2

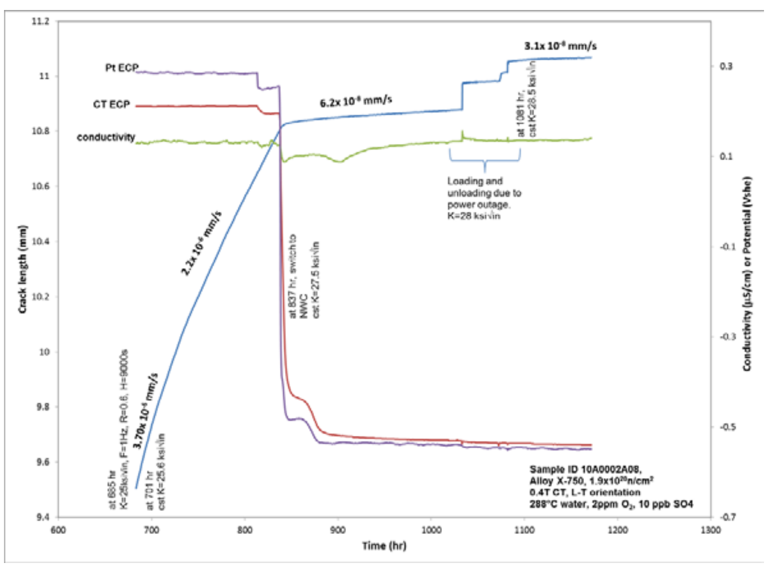


Location	Package	EPRI-3-1 Calculated Flux (n/cm^2-sec)	EPRI-3-2 Calculated Flux (n/cm^2-sec)	Calculated Fluence (n/cm^2)
Holder D				
D-4	TC	2.143E+13	1.851E+13	1.746E+20
D-3	TC	2.892E+13	2.497E+13	2.356E+20
D-2	TC	3.662E+13	3.162E+13	2.983E+20
D-1	TC	4.399E+13	3.799E+13	3.584E+20
Holder C				
C-10	EPRI-3C-8	6.494E+13	5.609E+13	5.291E+20
C-9	EPRI-3C-7	7.223E+13	6.238E+13	5.884E+20
C-8	EPRI-3C-6	8.050E+13	6.952E+13	6.558E+20
C-7	EPRI-3C-5	8.771E+13	7.575E+13	7.145E+20
C-6	NRC-3C-4	9.353E+13	8.077E+13	7.619E+20
C-5	NRC-3C-3	9.775E+13	8.442E+13	7.963E+20
C-4	NRC-3C-2	1.019E+14	8.797E+13	8.299E+20
C-3	NRC-3C-1	1.057E+14	9.125E+13	8.608E+20
C-2	EPRI-3B-13	1.063E+14	9.178E+13	8.658E+20
C-1	Spacer	***	***	***
Holder B				
B-14	EPRI-3B-14	1.120E+14	9.671E+13	9.123E+20
B-13	EPRI-3B-12	1.147E+14	9.908E+13	9.346E+20
B-12	EPRI-3B-11	1.164E+14	1.006E+14	9.487E+20
B-11	EPRI-3B-10	1.170E+14	1.010E+14	9.532E+20
B-10	EPRI-3B-9	1.177E+14	1.017E+14	9.590E+20
B-9	EPRI-3B-8	1.180E+14	1.019E+14	9.610E+20
B-8	EPRI-3B-7	1.195E+14	1.032E+14	9.734E+20
B-7	EPRI-3B-6	1.191E+14	1.029E+14	9.704E+20
B-6	EPRI-3B-5	1.182E+14	1.021E+14	9.629E+20
B-5	EPRI-3B-4	1.176E+14	1.016E+14	9.584E+20
B-4	EPRI-3B-3	1.171E+14	1.011E+14	9.538E+20
B-3	EPRI-3B-2	1.171E+14	1.011E+14	9.538E+20
B-2	EPRI-3B-1	1.153E+14	9.960E+13	9.395E+20
B-1	Spacer	***	***	***
Holder A				
A-12	Tensile-1	1.042E+14	8.999E+13	8.489E+20
A-12	Tensile-2	1.052E+14	9.087E+13	8.572E+20
A-12	Tensile-3	1.043E+14	9.005E+13	8.495E+20
A-12	Tensile-4	1.049E+14	9.057E+13	8.544E+20
A-11	Spacer	***	***	***
A-10	Spacer	***	***	***
A-9	Spacer	***	***	***
A-8	Spacer	***	***	***
A-7	Spacer	***	***	***
A-6	Spacer	***	***	***
A-5	Spacer	***	***	***
A-4	Spacer	***	***	***
A-3	Spacer	***	***	***
A-2	Spacer	***	***	***
A-1	Spacer	***	***	***

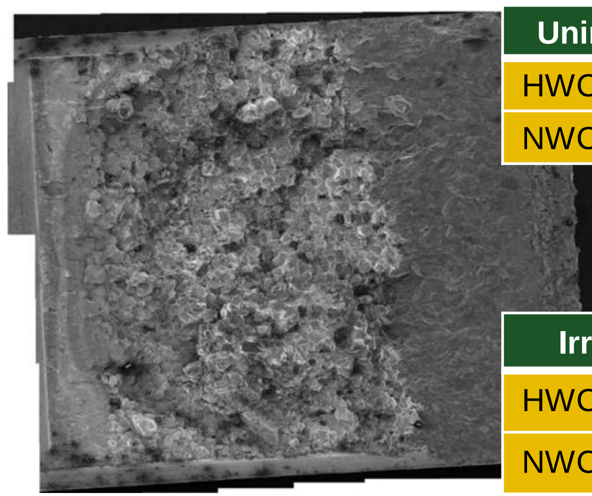
*** Flux in the spacers is not of interest regarding material test specimen PIE and is not reported.

IASCC second X-750 test

- **EPRI-2 target 2.0 X 10^{20} n/cm^2**
  - Actual fluence achieved1.93 X 10^{20} (damage ~0.283 dpa)
  - Irradiation temperature ~340°C
    - Combination of modeling and melt wire evaluation
    - Ensuing irradiations to employ T/Cs in test train
- **0.4T-CT, X-750 specimen in L-T orientation**



Sample IASCC results with transition from NWC to HWC



Post test SEM fractrograph

Unirradiated crack growth rate	
HWC	1.3 – 13 X 10^{-8} mm/s
NWC*	8.8 – 18 X 10^{-7} mm/s

* With 0-10 ppb sulfate

Irradiated crack growth rate	
HWC	3.1 – 6.2 X 10^{-8} mm/s
NWC	1.1 – 22 X 10^{-7} mm/s

K = 25 ksi-√in

Irradiated X-750 (HTH) (1.93×10^{20} n/cm^2) IASCC CGR Testing (confirmatory)

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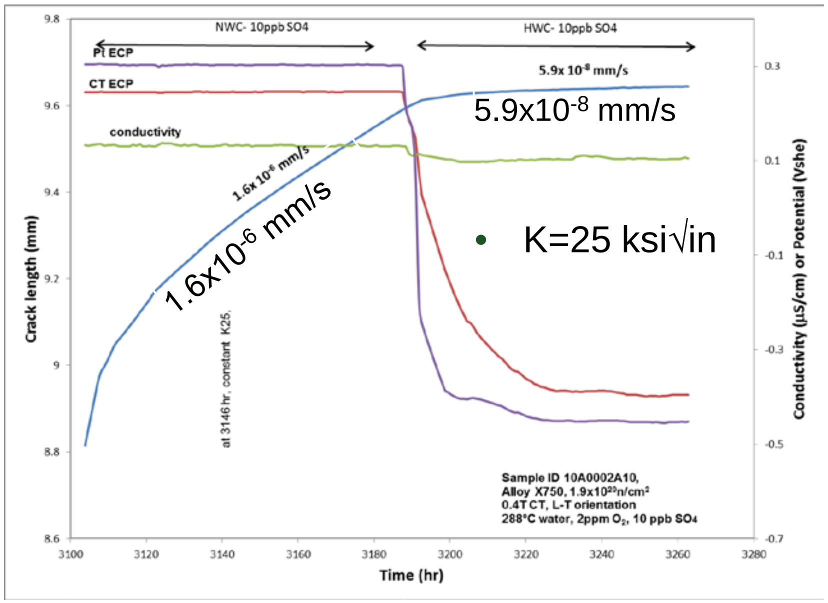
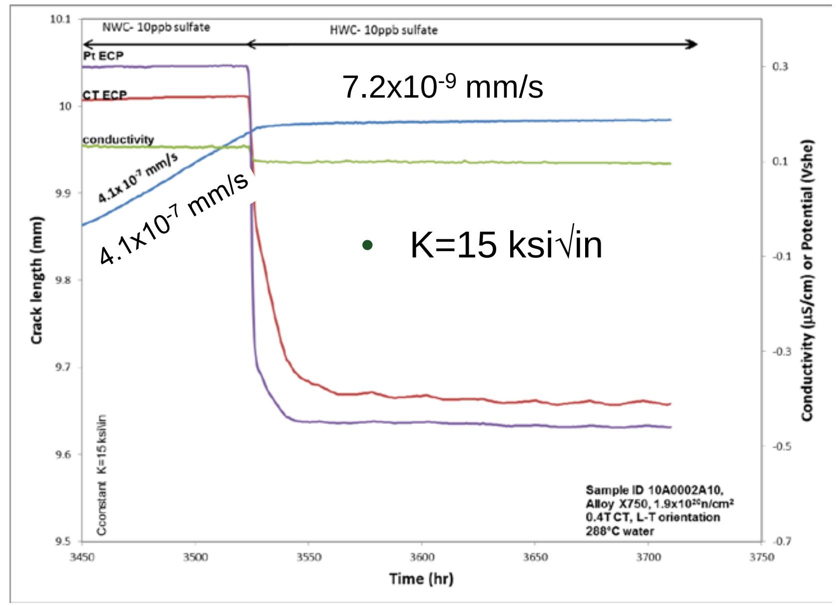
Confirmatory test in FY2016 with multiple K applied



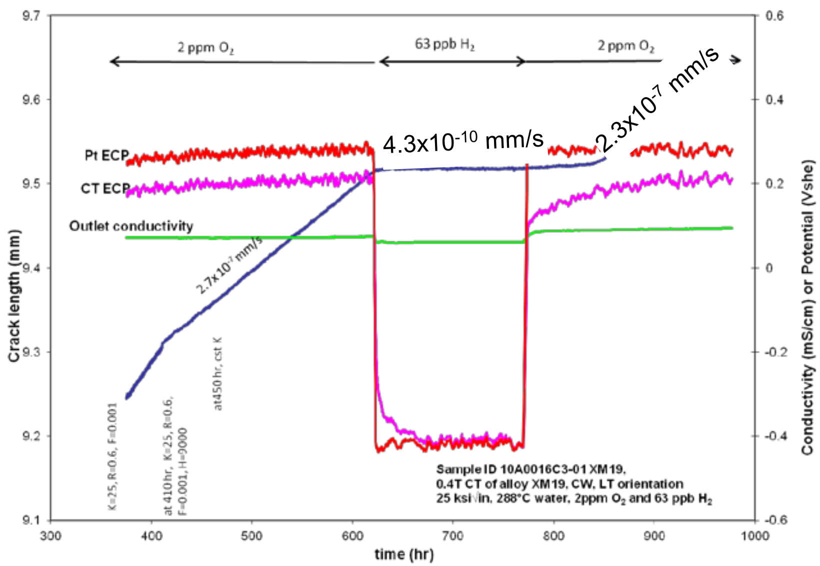


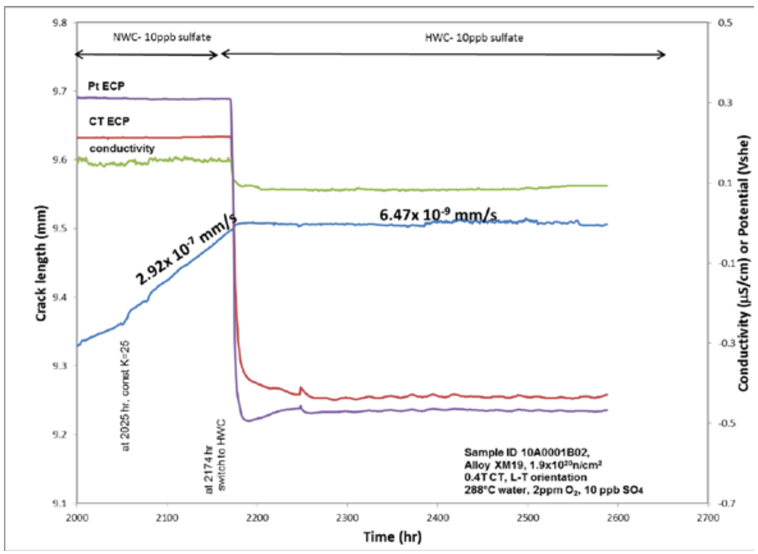
Irradiated crack growth rate*	
HWC	$3.1 – 6.2 \times 10^{-8}$ mm/s
NWC	$1.1 – 22 \times 10^{-7}$ mm/s

Irradiated crack growth rate*	
HWC	7.2×10^{-9} mm/s
NWC	4.1×10^{-7} mm/s

XM-19 SCC/IASCC testing

First IASCC test on EPRI-2 XM-19 completed in FY 2016





Unirradiated (19.3% CW) Crack Growth Rate	
NWC	2.6-3.1 x 10^{-7} mm/s
HWC	4.3X10^{-10} to 6.6X10^{-9} mm/s

Irradiated (1.9 X 10^{20} n/cm^2) Crack Growth Rate	
NWC	2.9 x 10^{-7} mm/s
HWC	6.5 x 10^{-9} mm/s

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EPRI-2 fracture toughness (X-750)

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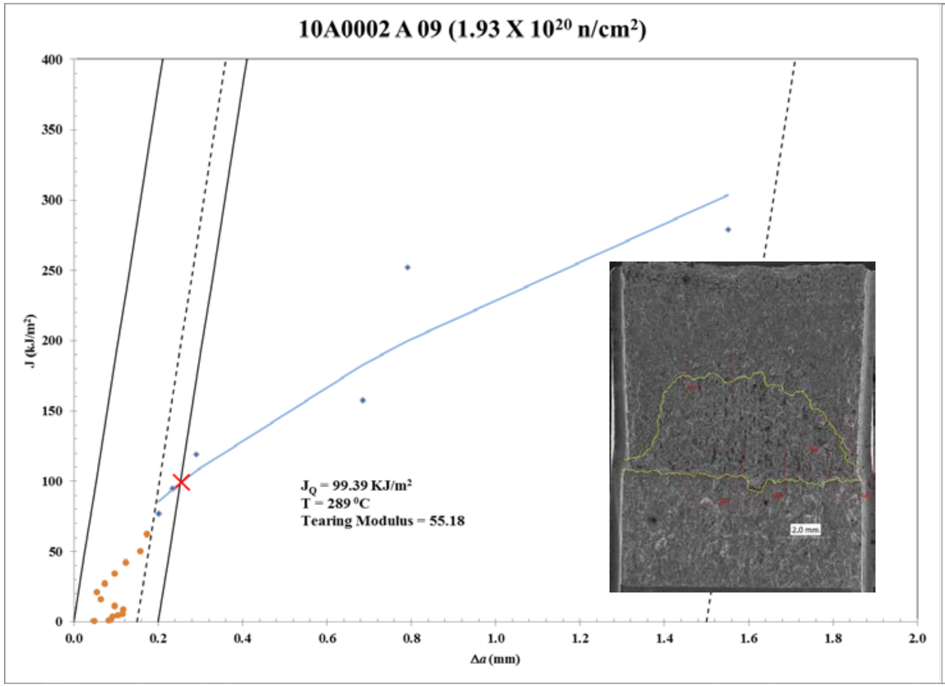
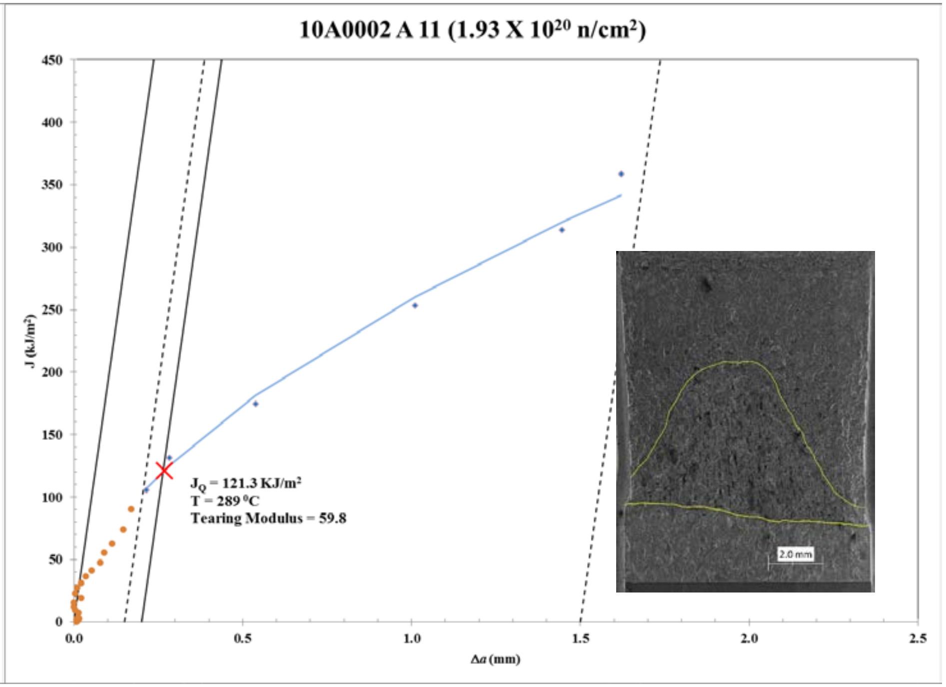
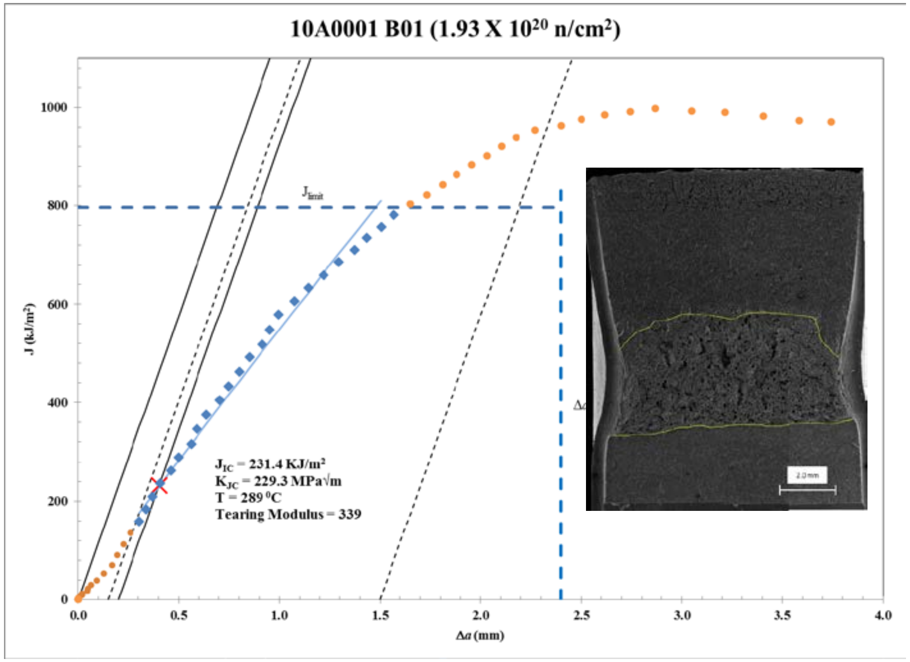
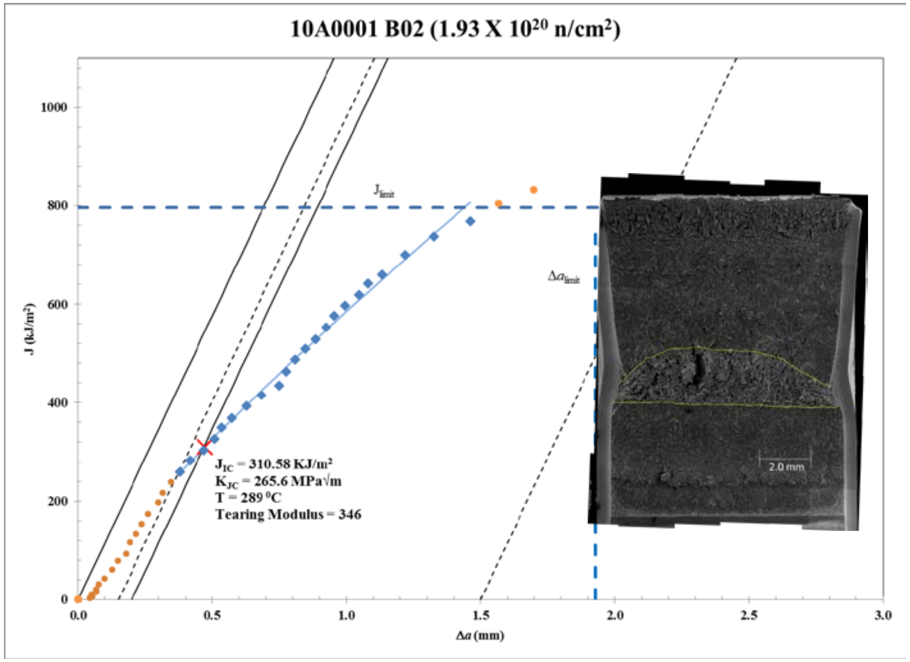




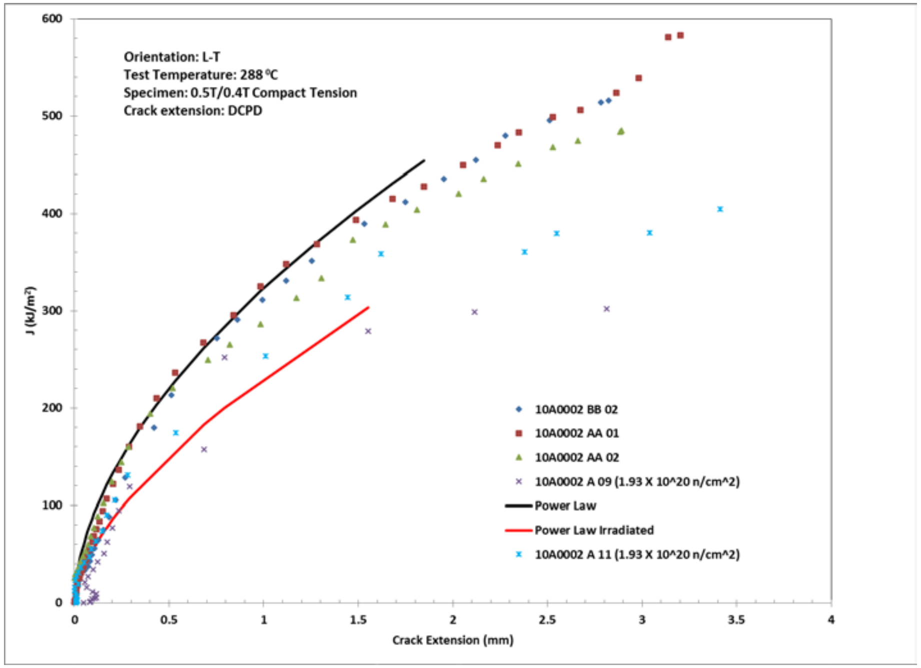
EPRI-2 fracture toughness (XM-19)



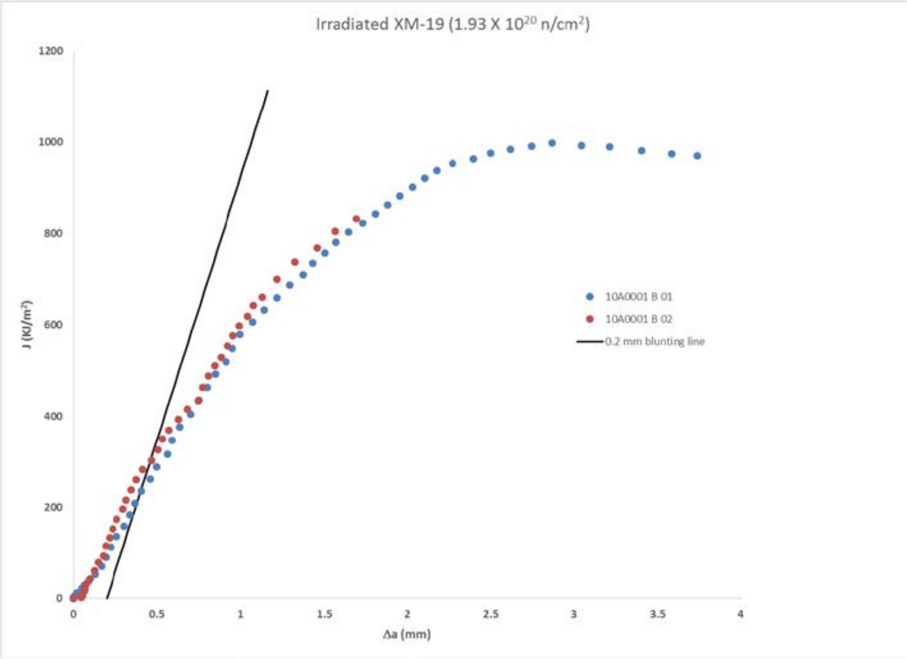


EPRI-2 Fracture toughness

X-750



XM-19



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2016 Accomplishments

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- **Irradiation of EPRI-3 capsule (1 X 10^{21} n/cm^2 target)**
  - 1st cycle completed April, 2014
  - 2nd cycle finished April, 2016
  - As-run estimated fluence ~ 0.96 X 10^{21} n/cm^2 (~1.5 dpa)
  - Issues with flow restriction through irradiation assembly
- **EPRI-1 and EPRI-3 received at MFC and catalogued**
- **Confirmatory IASCC CGR test of EPRI-2, X-750**
  - Confirms no effect of irradiation at this fluence
- **IASCC CGR test of EPRI-2 XM-19**
  - No effect of irradiation noted
- **Fracture toughness for EPRI-2, X-750 and XM-19 confirmatory**
  - Effect of irradiation is apparent for X-750
- **TEM analysis of EPRI-2 specimens**
  - New microscopist transitioned in FY2016

Pilot Project 2: EPRI Zirconium Growth Project

- **Experiment objectives: (fuel channel bowing issue)**
  - Measure hydrogen assisted, irradiation induced growth under BWR relevant conditions
  - Determine effects of hydrogen content (soluble vs. insoluble) and neutron fluence on growth strain
  - Characterize irradiation induced defects and correlate to macroscopic growth strain
- **Experiment strategy:**
  - Prepared 200 specimens; 35 mm long X 6.5 mm wide X 0.8 mm thick
  - 4 different Zr alloys (as received ~= 10 ppm H)
  - Pre-charged to H concentrations (~= 40 and 125 ppm)
  - Irradiate to 4 fluences and maintain temperature ~=285 C
  - Helium filled capsule for temperature control
  - Measure before and after irradiation using INL designed and built equipment

Zirconium Growth Irradiation

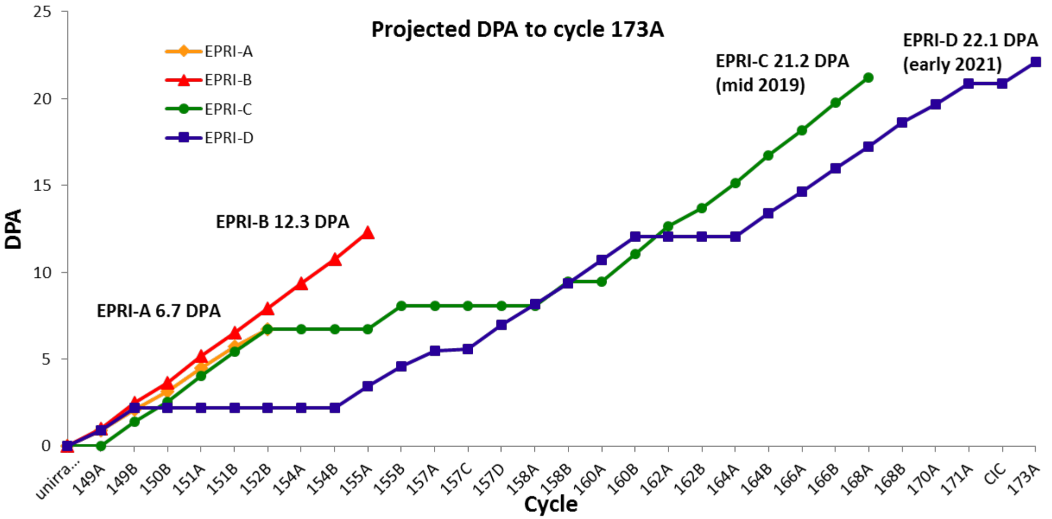
- **DPA estimates through cycle 158B (April 2016):**
  - EPRI A, 7 dpa target, 6.9 dpa actual
  - EPRI B, 10 dpa target, 12.3 dpa actual
  - EPRI C, 20 dpa target, 9.5 dpa actual (48% complete)
  - EPRI D, 30 dpa target, 9.4 dpa actual (31 % complete)

Zircaloy specimens

Capsule A - DPA at End of Irradiation

Position	Specimen Set	Cumulative DPA
A-16 (EPRI-A)	0 (bottom)	6.687
	1	6.871
	2	6.966
	3	7.023
	4	7.030
	5	7.063
	6	7.057
	7	7.022
	8	6.929
	9 (top)	6.680

Average (All Specimens): 6.933 DPA
Average (Specimen Sets 1-8): 6.995 DPA

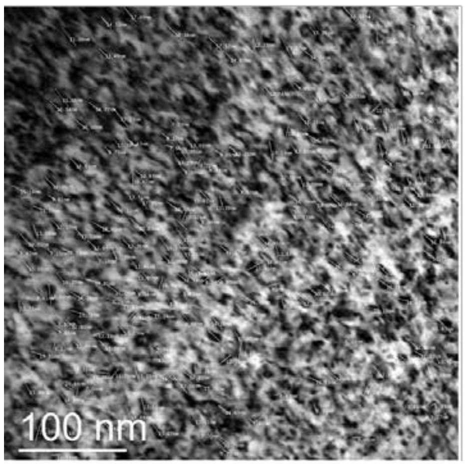


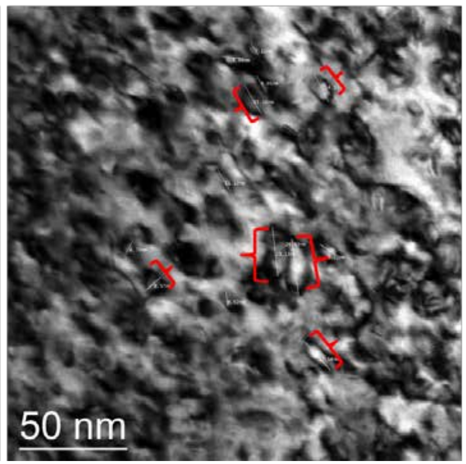
EPRI ZG TEM analyses

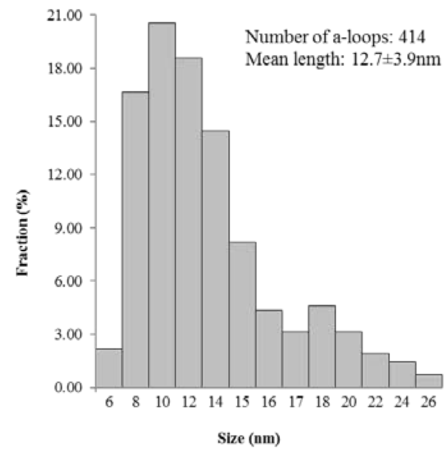
- TEM analyses initiated in early 2015 with specimen transfers
- Selection criteria – identify changes in:
  - Sn, Fe, and H content
  - Initial density of strain defects (cold work)
  - Radiation dose

11 Samples Selected for TEM	Capsule	Sample Identifier	Alloy Composition	Hydrogen Content
AR10z (12 dpa)	B	AR10Z-3	Zr-1%Nb-0.05%Fe	125 ppm
AR20z (12 dpa)	B	AR20Z-7	Zr-1%Nb-1%Sn-0.1%Fe	125 ppm
AWz (12 dpa)	B	AWZ-9	Zr-1%Nb-0.65%Sn-0.1%Fe	125 ppm
AGz (12 dpa)	B	AGZ-3	Zr-1%Nb-1%Sn-0.3%Fe	125 ppm
AR30x (12 dpa)	B	AR30X-2	Zircaloy-2	trace
AR30z (7dpa)	A	AR30Z-1	Zircaloy-2	125 ppm
AR30z (12 dpa)	B	AR30Z-10	Zircaloy-2	125 ppm
AAz(12 dpa)	B	AAZ-2	Zircaloy-4	125 ppm
AR36z (12 dpa)	B	AR36Z-4	Zircaloy-4	125 ppm
AR36y (12 dpa)	B	AR36Y-4	Zircaloy-4	40 ppm
AR36y (7 dpa)	A	AR36Y-7	Zircaloy-4	40 ppm
Additional 3 Samples if time allows	Capsule	Sample Identifier		
BWz (12 dpa)	B	BWZ-5	Zr-1%Nb-0.65%Sn-0.35%Fe	125 ppm
AR10z (7 dpa)	A	AR10Z-2	Zr-1%Nb-0.05%Fe	125 ppm
BGz (12 dpa)	B	BGZ-8	Zr-1%Cr-0.5%Sn-0.5%Fe	125 ppm

EPRI ZG TEM analyses (<a> and <c> loop analyses)

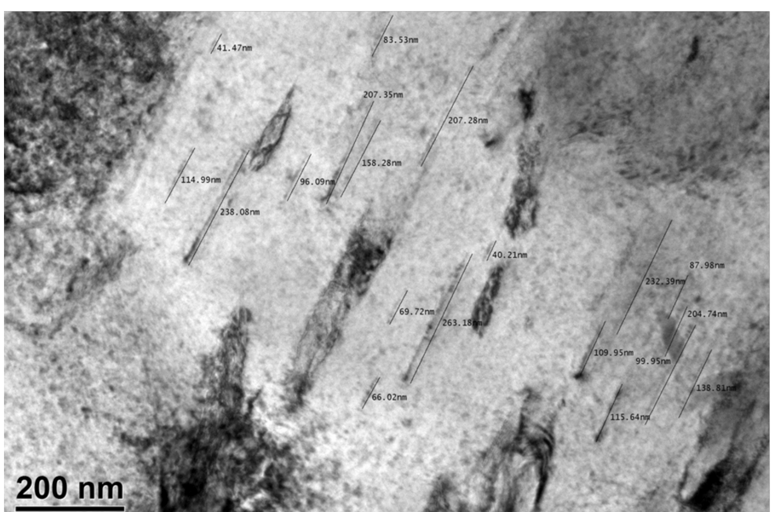


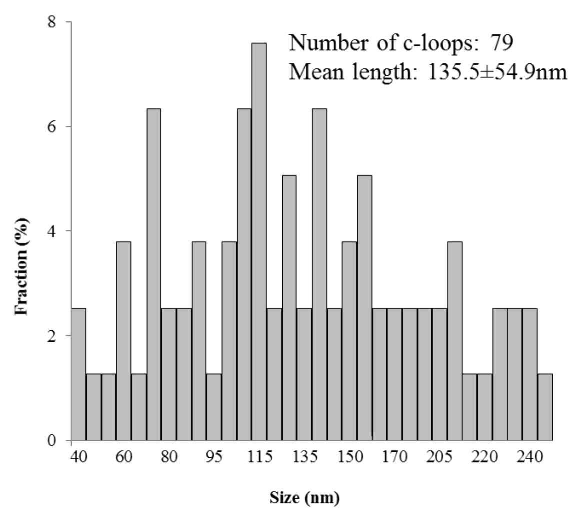




Sample AWZ-9

Loop density was calculated using linear density method. Estimated a-loop density is 1.37×10^{-4} loops/nm^2





Sample AR30Z-10

Loop density was calculated using linear density method. Estimated c-loop density is 1.12×10^{-5} loops/nm^2

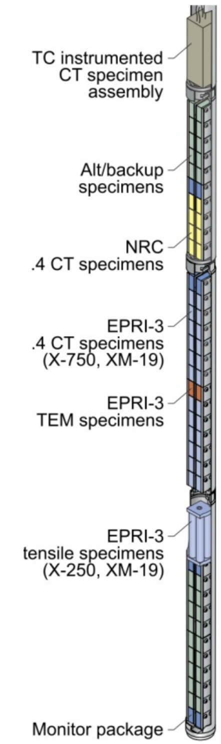
Zirconium Growth project progress

- **EPRI-C and EPRI-D irradiations continuing in ATR, DPA lagging due to lobe power outside of ±1 MW power band requested by EPRI**
- **TEM analyses on select EPRI-A and EPRI-B specimens continued in 2016.**
  - New microscopist hired (Daniel Jadernas – Studsvik)

		Irradiation-induced defect analysis				
Sample ID	Dose (dpa)	Imaging of defects	<a> or <c> loop determination	Loop density	FIB sample analysis	Loop size distribution
AWZ-9	12	Completed	Completed	Completed	2016	Completed
AR10Z-3	12	Completed	Completed	Data analysis ongoing	Completed	Data analysis ongoing
AR30Z-10	12	Planar sample characterization needed	Completed	Completed	Completed	Completed
AGZ-3	12	Planar sample characterization needed	Data analysis ongoing	Data analysis ongoing	Completed	Data analysis ongoing
AAZ-2	12	Ongoing	Ongoing	Ongoing	2016	Ongoing
AR30Z-1	7	2016	2016	2016	2016	2016
AR36Z-4	12	2016	2016	2016	2016	2016
AR36Y-4	12	2016	2016	2016	2016	2016
AR36Y-7	7	2016	2016	2016	2016	2016
AR20Z-7	12	2016	2016	2016	2016	2016
AR30X-2	12	2016	2016	2016	2016	2016

NRC: Irradiation and PIE of 304 SS

- **Experiment objectives:**
  - Determine effect of neutron irradiation on sensitized 304 and 304L weld HAZ
  - Measure fracture toughness and CGR under BWR conditions
    - Normal Water Chemistry
  - Validate INL capability for irradiation and PIE of austenitic SS
  - Provide additional data point for CGR as a function of fluence
- **Experiment strategy:**
  - Prepared 12 specimens
    - 6 of each
    - 2 of each used for baseline CGR and FT
    - 4 of each irradiated along with EPRI-3 test capsule
    - 2 of each tested at INL, and 2 of each tested at ANL
- **Completed irradiation in 2016**
  - Testing to be initiated



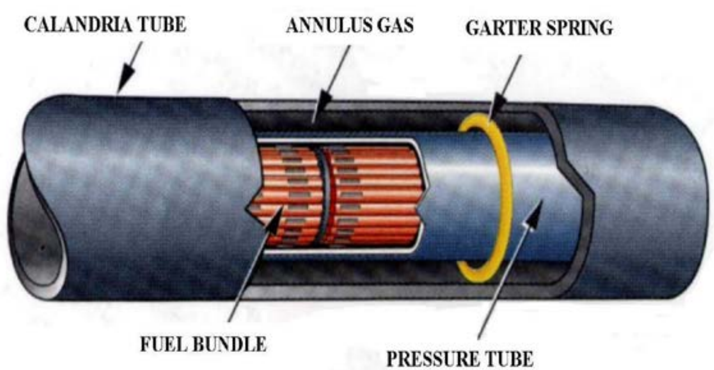
AECL(CNL): Characterization of X-750 from CANDU reactor

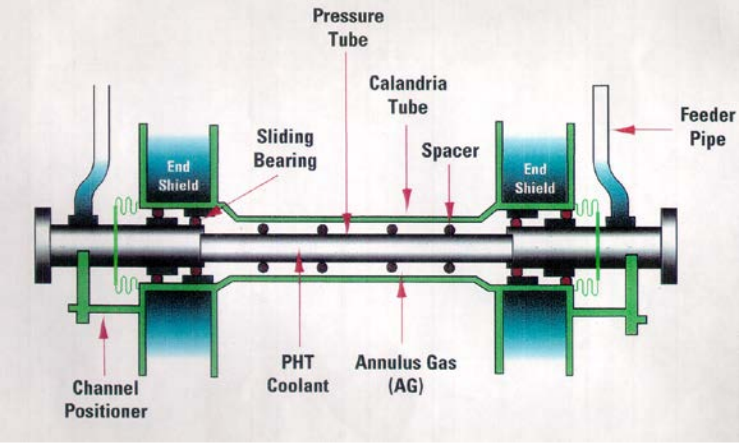
Experiment objectives:

- Determine embrittlement mechanism for X-750 in calandria tube springs
- Use hot FIB to fabricate TEM lamellae and observe microstructure
- Correlate microhardness measurements to microstructural observations

Experiment strategy:

- Specimens extracted from different locations on spring and prepped at AECL
- Shipped to INL and sectioned for examination





U.S. DEPARTMENT OF ENERGY

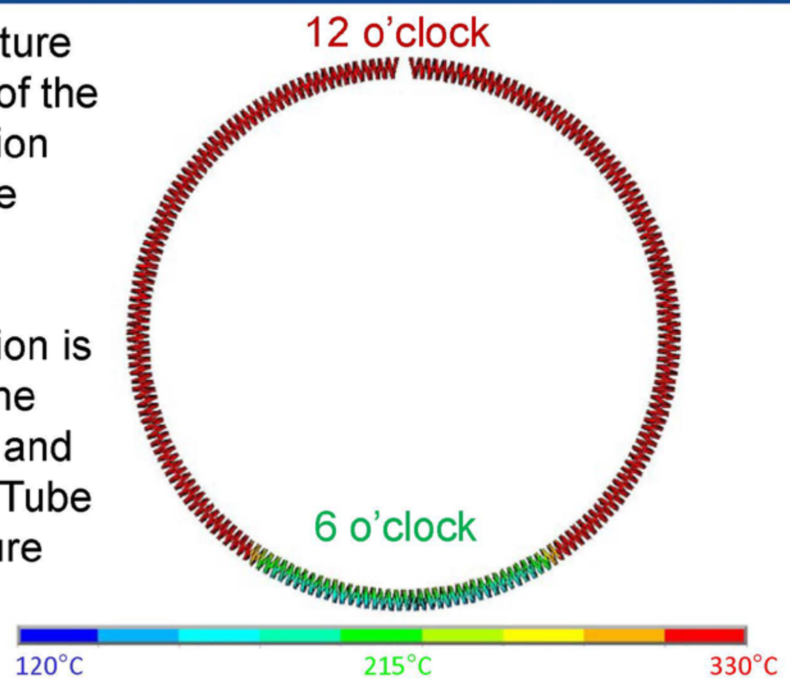
Nuclear Energy

AECL project details

Nuclear Science User Facilities

Operating Temperatures

- Irradiation temperature varies as a function of the circumferential location (clock position) of the garter spring on the Pressure Tube.
- The 6 o’clock position is “pinched” between the “hot” Pressure Tube and the “cold” Calandria Tube creating a temperature gradient in the component.



AECL EACL

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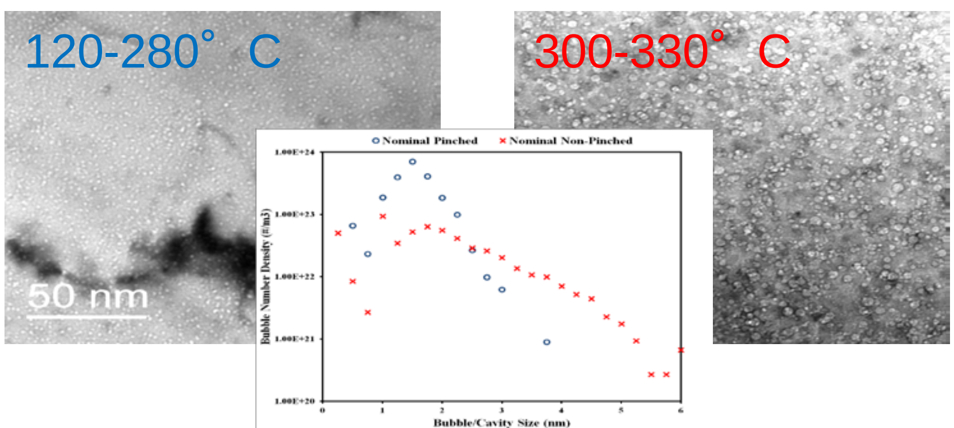
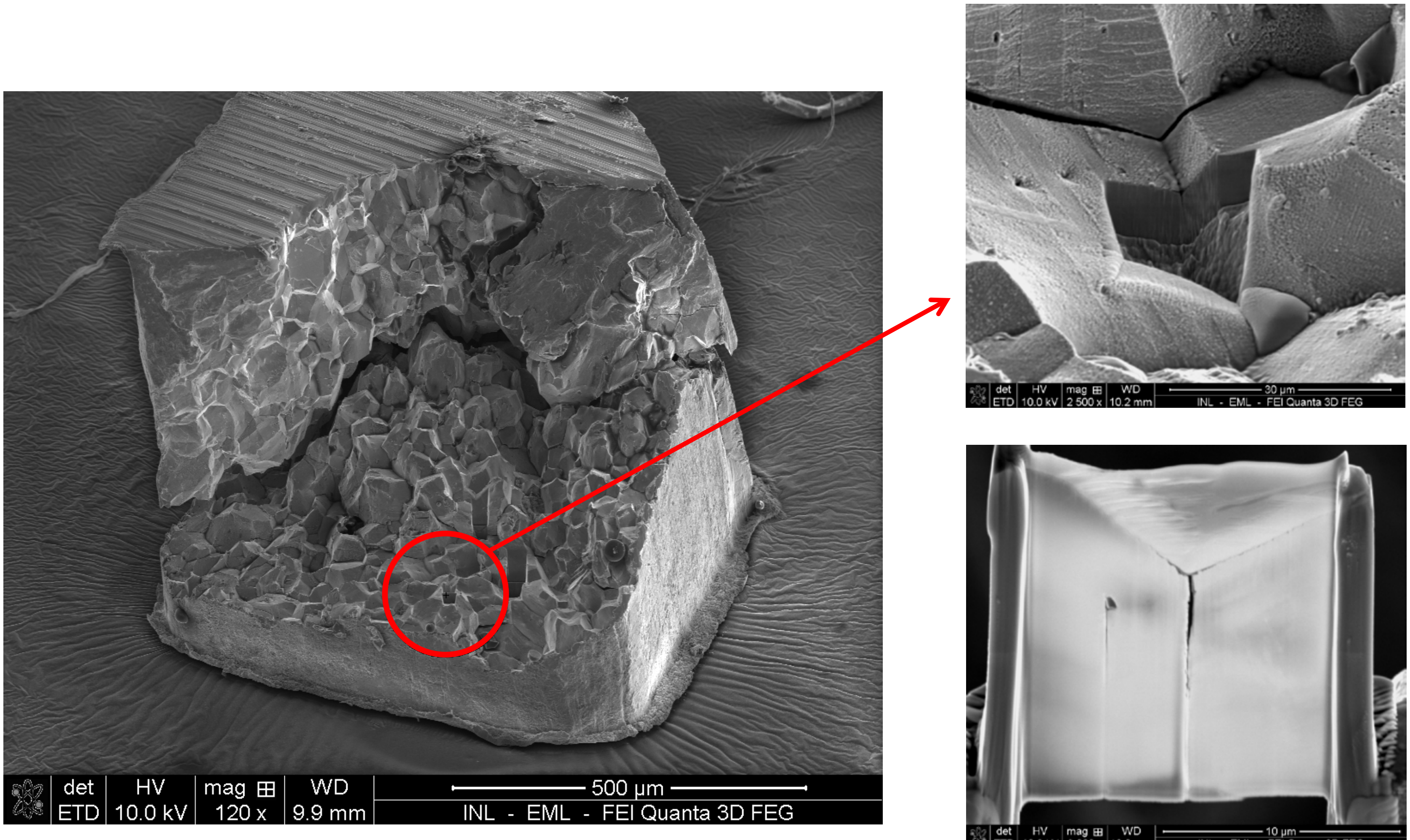
TEM examination

- **The focus of the examination has been primarily on:**
  - He bubbles
  - Stability of secondary gamma prime precipitates
  - Grain boundary microstructure evolution
- **During the examination, advanced electron microscopy techniques have been employed to give a broader understanding of the material degradation.**
  - TEM tomography of helium bubbles
  - Electron energy loss spectroscopy of helium within nano-bubbles

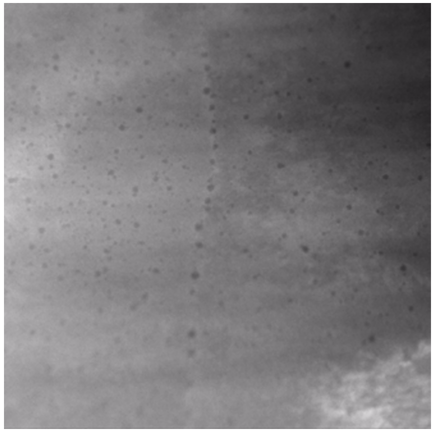


TEM examination of intergranular fracture



CNL X-750 analysis conclusions

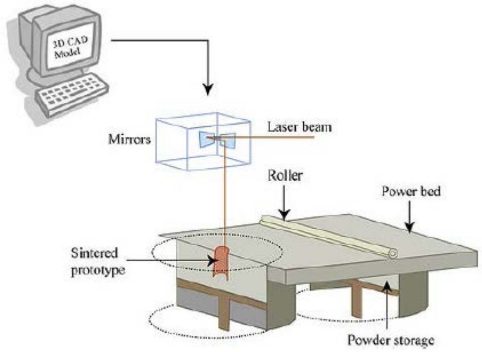
- **Intergranular fracture of Inconel X-750 has been directly linked to grain boundary helium bubbles.**
- **The microstructural evolution is directly linked with the irradiation temperature of the component.**
  - Bubble size and density
  - Gamma prime disordering
- **Microhardness has confirmed that although the component has reduced ductility and load carrying capacity, evaluation of available microhardness data shows no conclusive evidence that the yield strength of the material has changed from the unirradiated state.**

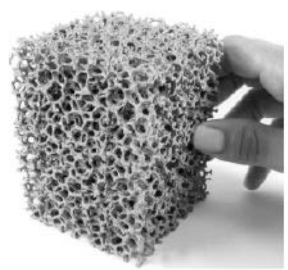


Tomography (Grain Boundary He Bubbles)

CINR award for 2017 – GE-Hitachi (NSUF access only)

- **Irradiation Testing of LWR Additively Manufactured Materials**
  - Direct Metal Laser Melting (DMLM) processed
- **Objectives**
  - Irradiate and characterize additively manufactured 316L and IN 718
  - Compare data to similar data obtained in wrought material
  - Extend knowledge base from unirradiated characterization
- **NSUF Facilities**
  - ATR, EIL SCC, IASCC, EML
- **Address fundamental knowledge gap**
  - No data on AM materials in prototypical LWR conditions





Ref: UTEP

Ref. Within Labs, UK

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