U.S. DEPARTMENT OF ENERGY | Nuclear Energy

*Nuclear Science User Facilities*

# Industry Program update 2015

## (INL/MIS-14-32147) Rev 3

**John H. Jackson**
Industry Program Lead

FY 2015 NSUF Annual Program Review
Germantown, MD
March 1-2, 2016

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# Engaging Industry

NSUF Nuclear Science User Facilities

- **A(nnual) NSUF Industry Advisory Committee (ANIAC)**
  - Direct industry participation in goal setting and strategy
- **Priorities in development**
  - Development of IASCC test capability was a priority at INL
- **Interaction with academia through NSUF as a hub**
  - Network of university and laboratory partners
  - Leverage strengths from all sides
- **Can propose directly for access to facilities (no university partner required)**
  - Very positive feedback from industry
  - Three facility access only proposals from industry in 2016

# ANIAC 2015



- **Hosted by EPRI Primary Systems Corrosion Research group**
  - Discussion of high priority items in EPRI Issue Management Tables
  - Specific areas of crossover with DOE mission
    - For example, IASCC, cable ageing, concrete ageing, accident tolerant fuels.
  - Use IMT crossover as guidance for CINR FOA
- **25 attendees**
  - EPRI, NSUF, DOE-NE, NRC, CNL, Westinghouse, UM, UCSB, BSU, Exelon, ***NuScale**, ***Terrapower**, Rolls Royce, ORNL, ATI Consulting
- **Specific feedback**
  - An NSUF symposium should be explored at a materials conference (TMS, etc)
  - Increase focus on fuels related projects
  - International collaboration should continue and be enhanced
  - Industry presence at NSUF Users Meeting should be encouraged
  - Instrumentation for irradiation experiments should be improved

# 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**

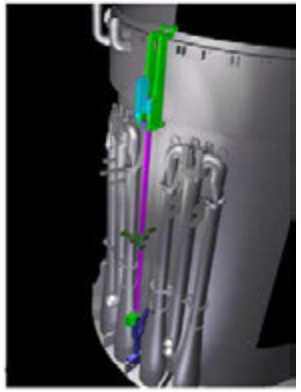
# Current Industry Programs

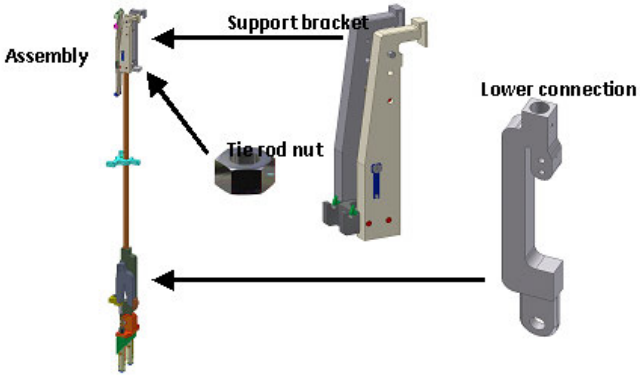


- **EPRI Pilot Project: Irradiation and PIE of alloys X-750 and XM-19 (three phases)**
  - Irradiate to three fluences (5 X $10^{19}$, 2 X $10^{20}$, 1 X $10^{21}$ n/cm$^2$) at ATR
  - Perform tensile, CGR, FT tests, and TEM microstructural analyses
- **EPRI Pilot Project: Zirconium alloy irradiation growth**
  - Measure hydrogen assisted irradiation induced growth under BWR relevant conditions.
  - Characterize irradiation induced defects and correlate to growth
- **AECL(CNL): TEM and microhardness of Candu reactor springs**
  - Alloy X-750 garter springs
  - FIB and TEM of highly irradiated spring sections (between 5 and 55 dpa)
  - **Finished in 2015!**
- **NRC: Irradiation and testing of austenitic SS**
  - Sensitized 304 and 304L weld HAZ
  - Piggyback with EPRI specimens

# 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**





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# 2015 Accomplishments

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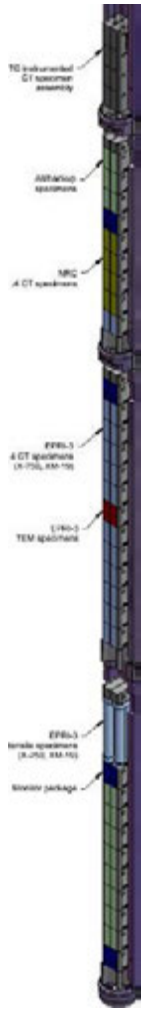
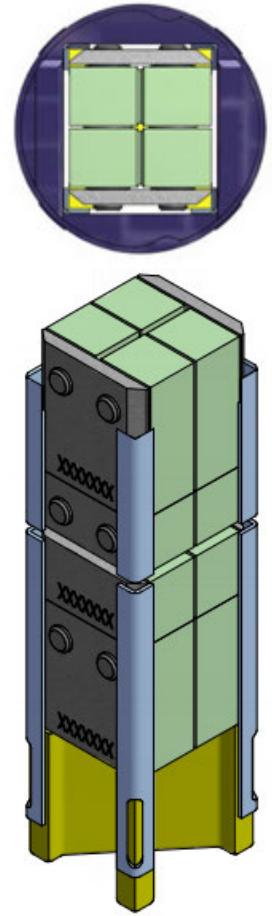
- **Irradiation of EPRI-1 capsule (5 X $10^{19}$ n/cm$^2$ target)**
  - Cycle 157C-2, 5.4 day cycle (February 12-17, 2015)
  - As-run estimated fluence ~5.2 X $10^{19}$ n/cm$^2$
  - Integrated thermocouples allowed more precise temperature control
  - Implemented flow restriction mitigation and centering mechanism
- **Fracture toughness for EPRI-2, X-750 and XM-19**
  - April, 2015
  - First fracture toughness tests to be completed using new IASCC systems
- **Tensile tests for EPRI-2, X-750 and XM-19**
  - June, 2015
  - 1$^{st}$ tensile tests using HFEF Instron
- **TEM analysis preparation**
  - Materials transferred and polished
  - Scheduled for early 2016

# Loop 2A flow restriction mitigation



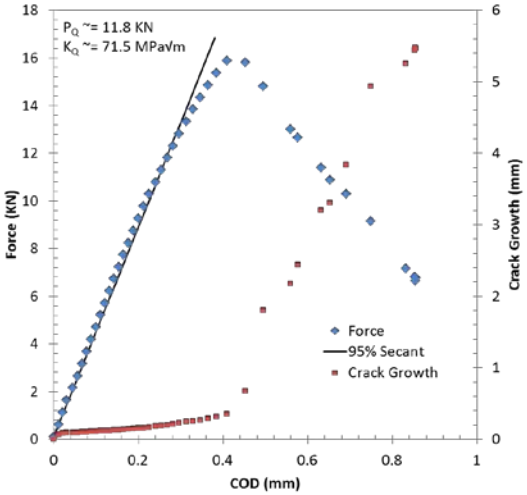
Capsule holder “A” replaced with hydraulic and thermal equivalent



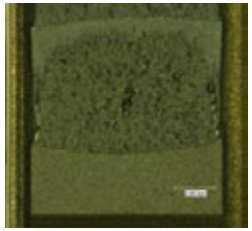
Alignment system to prevent package “twisting”

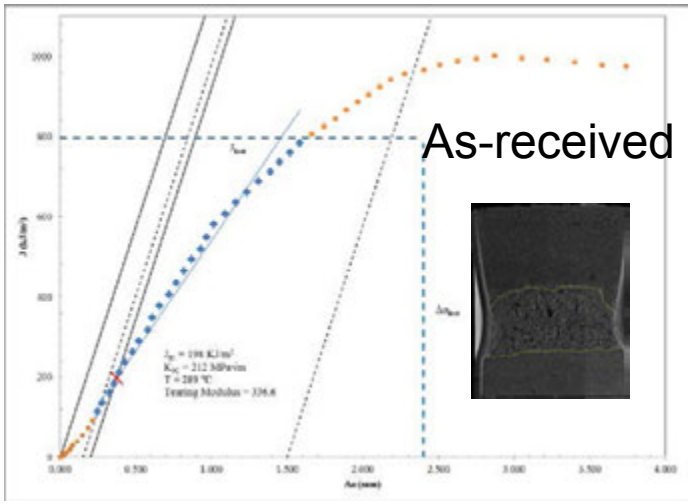
- Implemented in EPRI-1 in early 2015
- To be implemented in EPRI-3 in 2016

# EPRI-2 fracture toughness



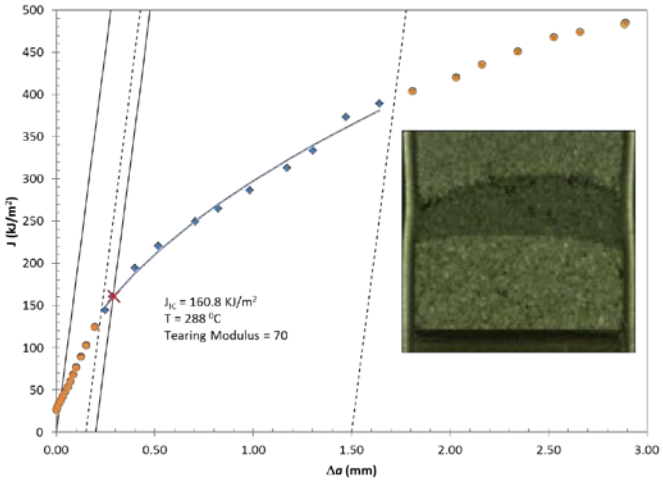

19.3% CW



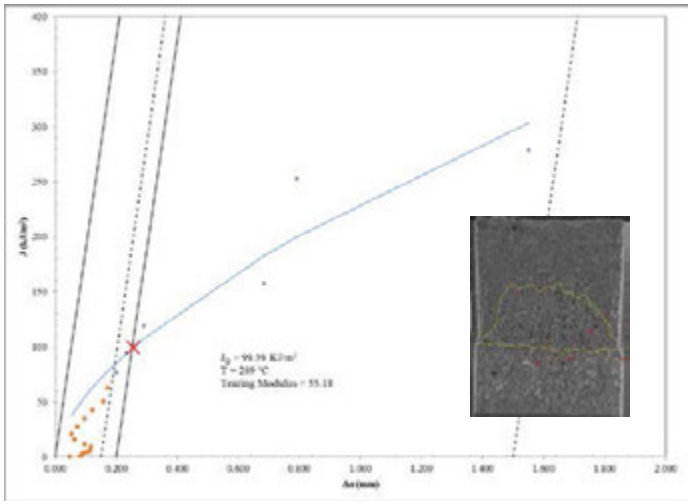



XM-19

Baseline



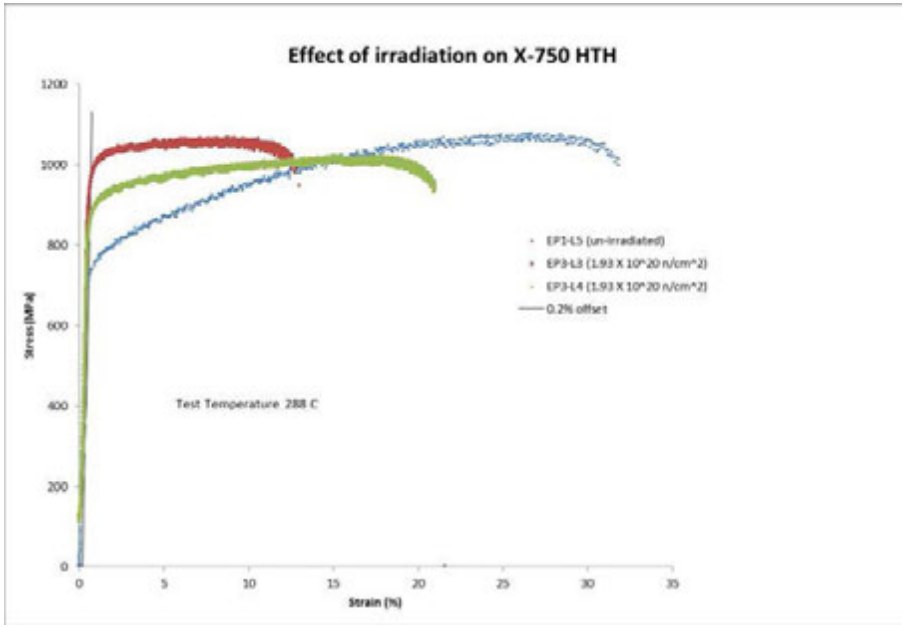

1.93 X $10^{20}$ n/cm$^2$

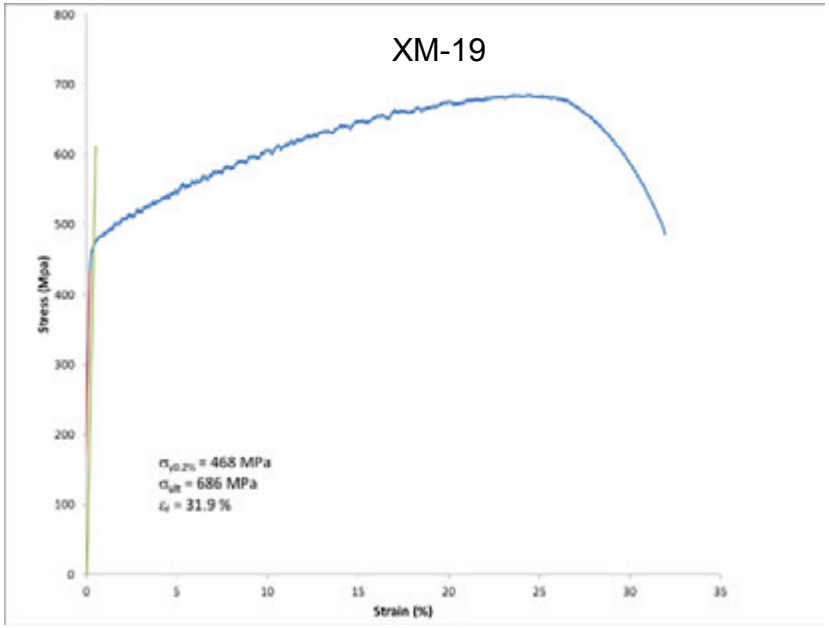


X-750

# EPRI-2 Tensile Tests

- **Tensile tests on X-750 and XM-19 (1.93 X $10^{20}$ n/cm$^2$)**
- **One XM-19 tensile specimen ruined during setup**



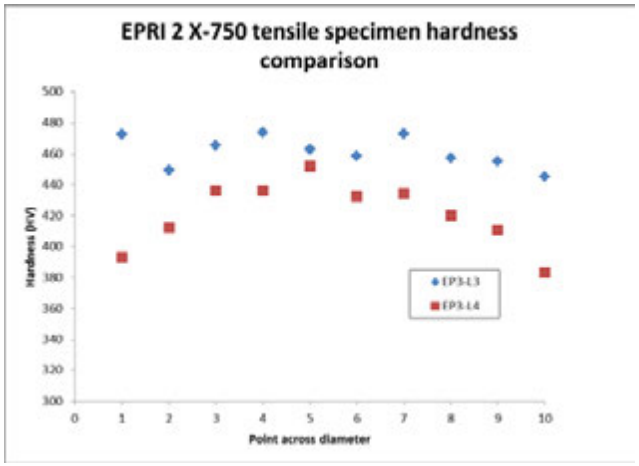

| 1.93 X $10^{20}$ n/cm$^2$ | 0.2% Offset Yield | Ultimate Tensile Strength | Ductility |
|---|---|---|---|
| | (MPa) | (MPa) | (%) |
| XM-19-04(SAMEPRI2B11_4) | 468 | 686 | 32 |
| EP3-L3 (SAMPERI2B11_1) | 962 | 1067 | 13 |
| EP3-L4(SAMEPRI2B11_2) | 877 | 1023 | 21 |

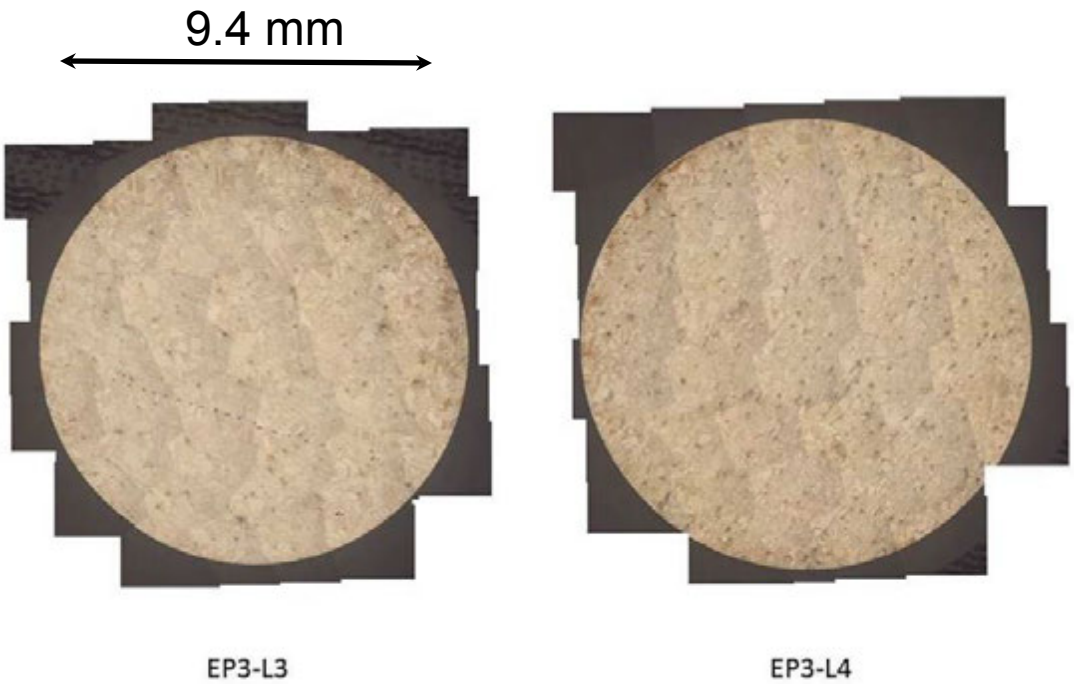
Baseline (un-irradiated)

- X-750 – $\sigma_y$ = 715 MPa, $\sigma_{ult}$ = 1082 MPa, $\varepsilon_f$ =30%
- XM-19 – $\sigma_y$ = 250 MPa, $\sigma_{ult}$ = 620 MPa, $\varepsilon_f$ = 40%

# EPRI-2 Tensile

- **X-750 further examination**
  - Cross section ends of both tensile specimens



# 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

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# Zirconium Growth project progress

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- EPRI-A irradiation complete, strain measurements complete, as-run DPA analysis complete, temperature analysis complete
- EPRI-B irradiation complete, strain measurements completed August 2014, temperature analysis ongoing (SiC monitors)
- TEM and micro-structural characterization of zirconium ongoing, analysis of specimen preparation methods using spare Zr specimens
- 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 commenced in late 2015.

# Zirconium Growth Irradiation

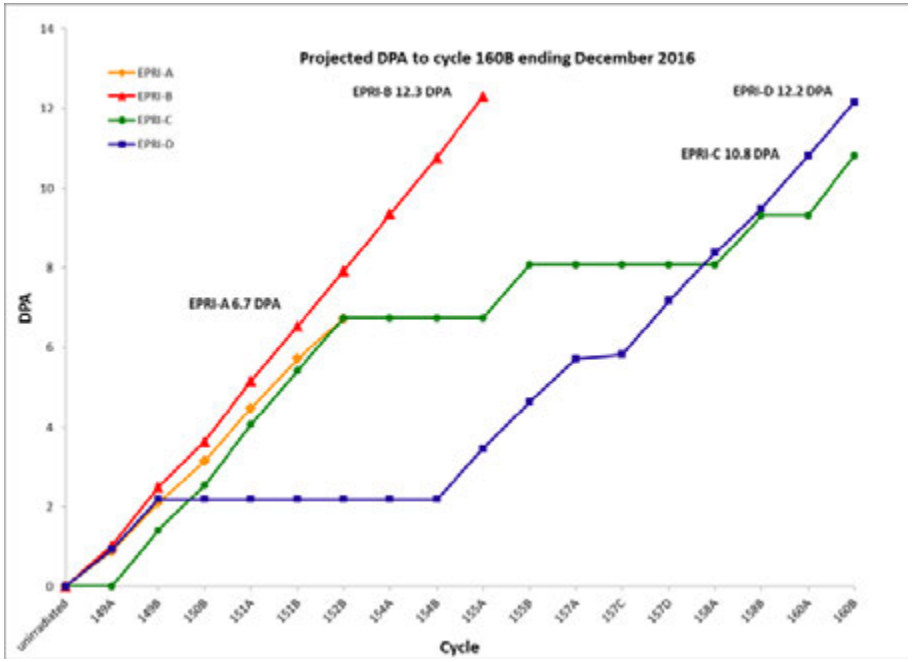
- **DPA estimates through cycle 155B:**
  - EPRI A, 7 dpa target, 6.9 dpa actual
  - EPRI B, 10 dpa target, 12.3 dpa actual
  - EPRI C, 20 dpa target, 8.1 dpa actual (41% complete)
  - EPRI D, 30 dpa target, 4.6 dpa actual (15 % complete)

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

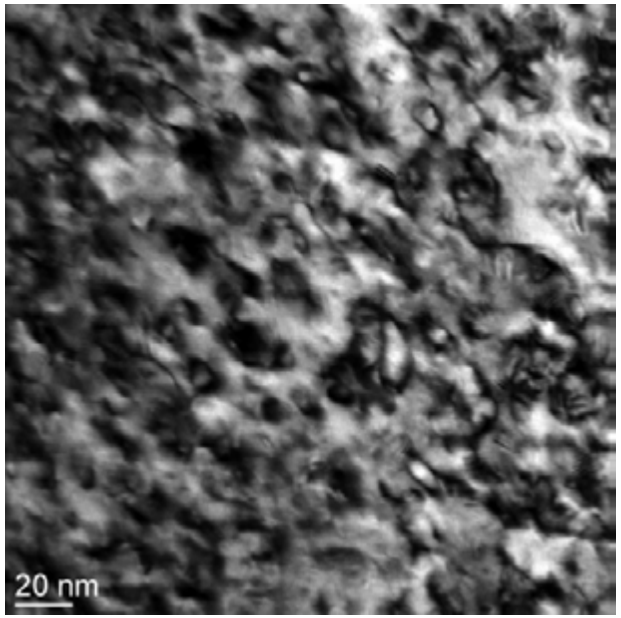


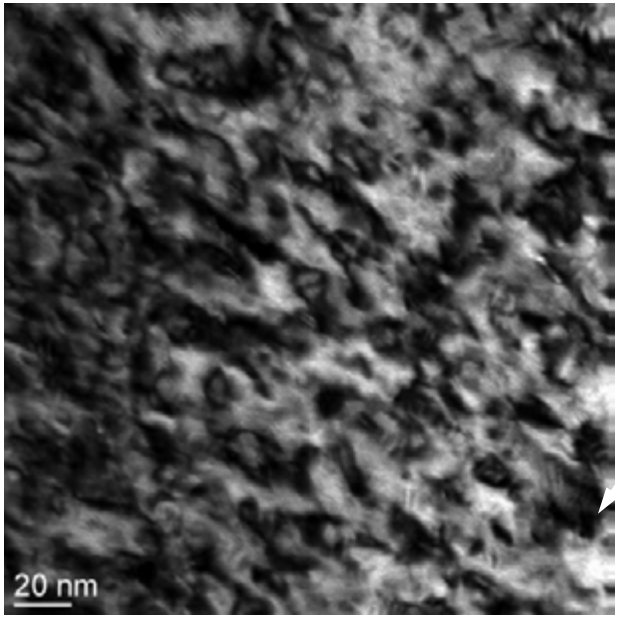
# EPRI ZG TEM analyses

- **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

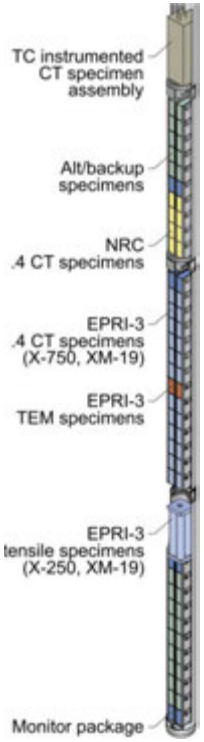



Bright-field transmission electron (TEM) micrographs showing <a> component dislocation loops in Zr alloy irradiated to 12 dpa at 280C. a) g=01-1-1, b) g=0-111 (as indicated by white arrows). High density of a-type network dislocations is observed at this low temperature irradiation.

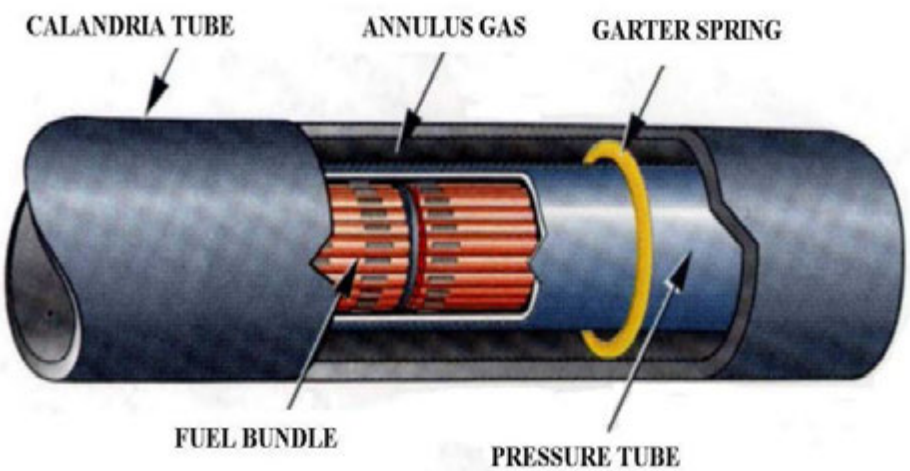
# NRC: Irradiation and PIE of 304 SS

- **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



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

- **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

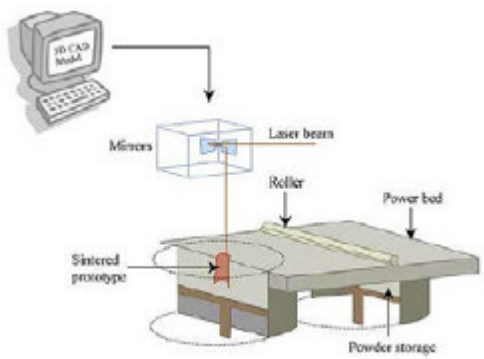


# CINR proposal for 2016 – PNNL/EPRI (NSUF access only)

- **Proposal**
  - Study Synergistic Effects of Thermal and Neutron Irradiation Aging on the Degradation of Cast Austenitic Stainless Steels (CASS) and stainless steel welds
- **Objectives**
  - Understand the mechanisms related to the combined effects of thermal and neutron irradiation aging on CASS and stainless steel welds
  - Determine the synergistic effects on fracture toughness and IASCC for CASS and SS welds
- **NSUF facilities**
  - ATR, IASCC, CAES, PNNL RPL and PNNL MSTL
- **Address high priority R&D needs and knowledge gaps:**
  - FM-RFA (Fundamental Materials Research Focus Area): I-05 → the effects of irradiation aging or synergetic aging on fracture toughness
  - PWR IMT Gap: P-AS-13a → Synergistic Effects on CASS
  - PWR IMT Gap: P-AS-13b → Synergistic Effects on SS welds

# CINR proposal for 2016 – 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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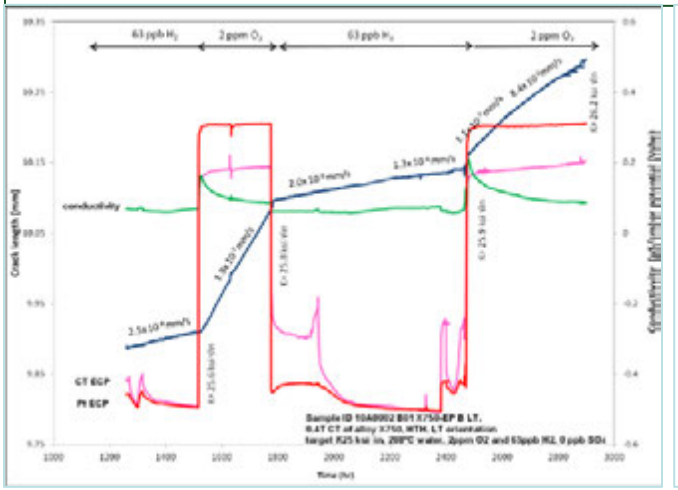
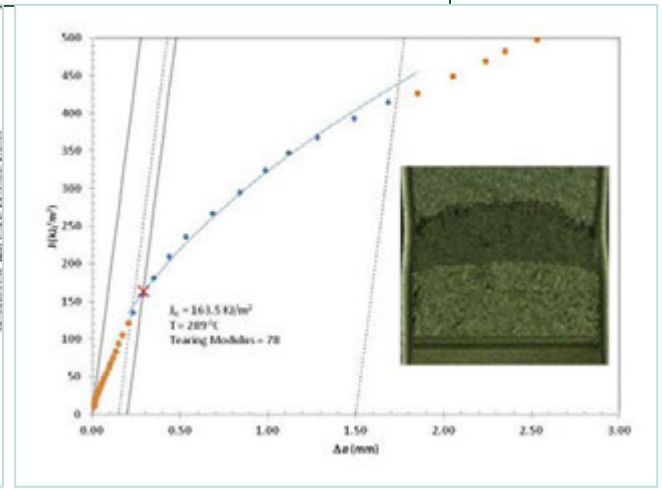
# Back up slides

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

- 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

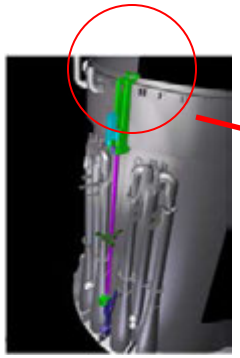


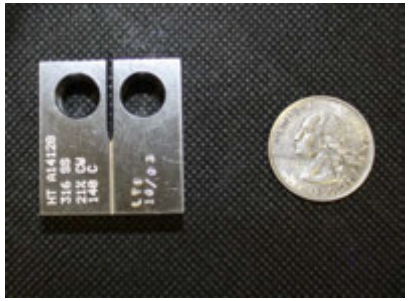

SCC of un-irradiated inconel X-750




Fracture toughness of un-irradiated X-750

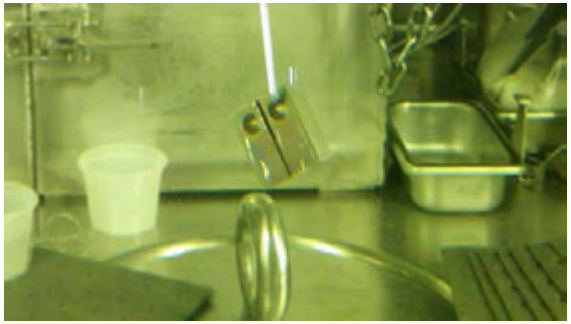
IASCC test systems

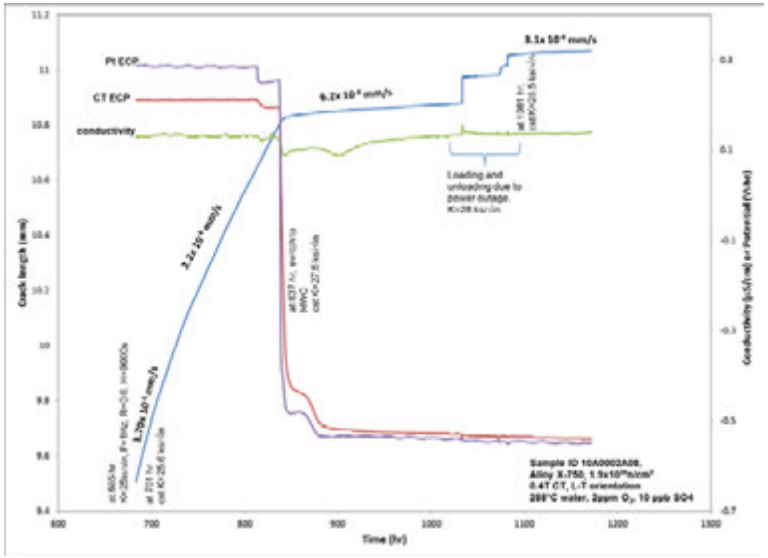




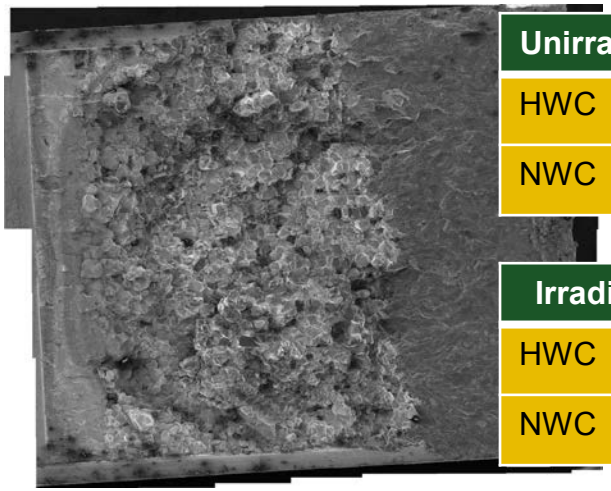
# IASCC first results (X-750)

- **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



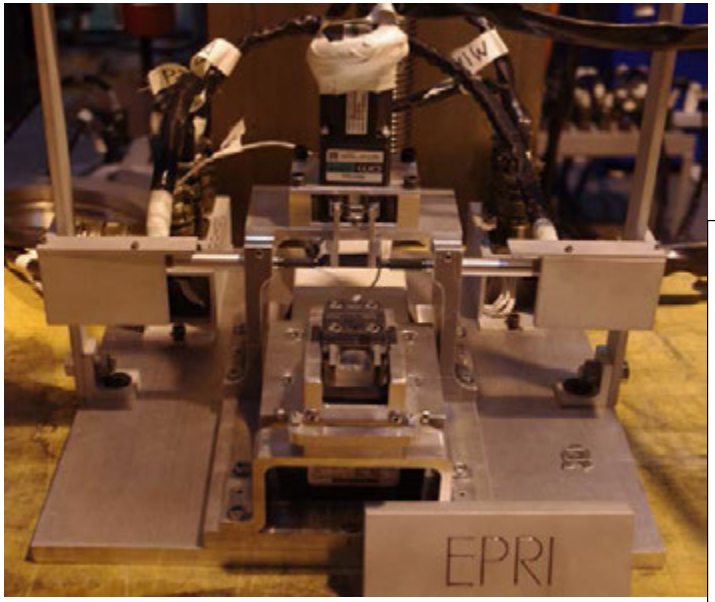
Post test SEM fractrograph

| Unirradiated crack growth rate | |
|---|---|
| HWC | 1.3 – 5.0 X $10^{-8}$ mm/s |
| NWC | 1.2 – 8.0 X $10^{-7}$ mm/s |

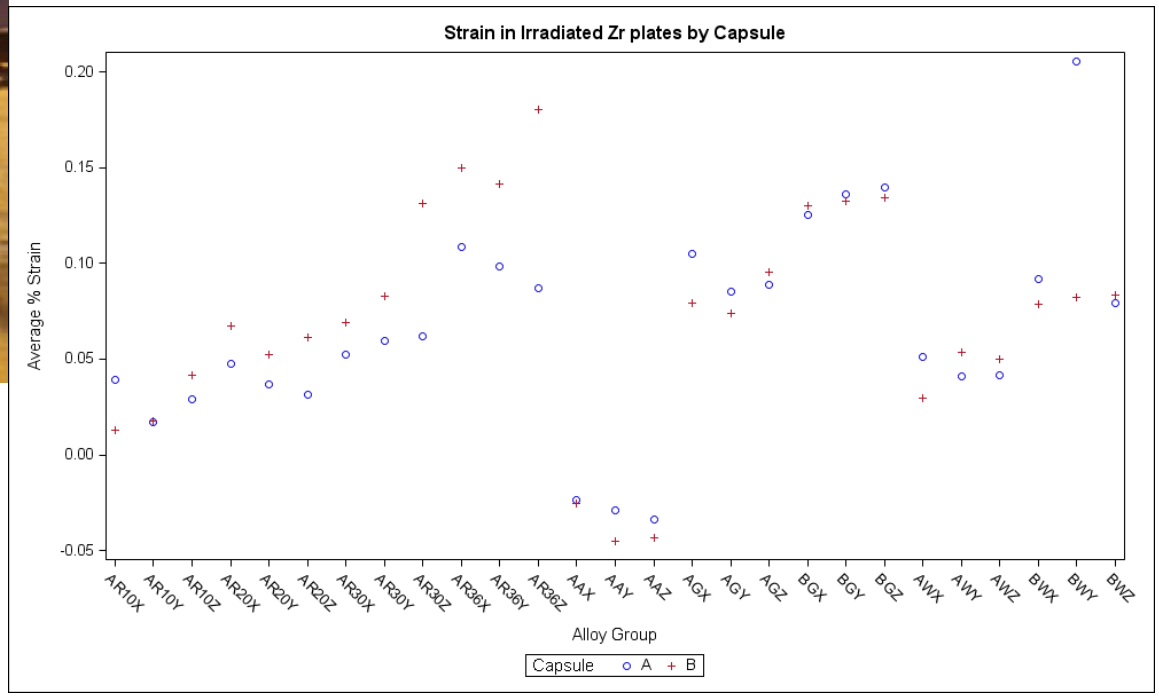
| Irradiated crack growth rate* | |
|---|---|
| HWC | 3.1 – 6.2 X $10^{-8}$ mm/s |
| NWC | 1.1 – 22 X $10^{-7}$ mm/s |

*Preliminary

# Zirconium Strain measurements



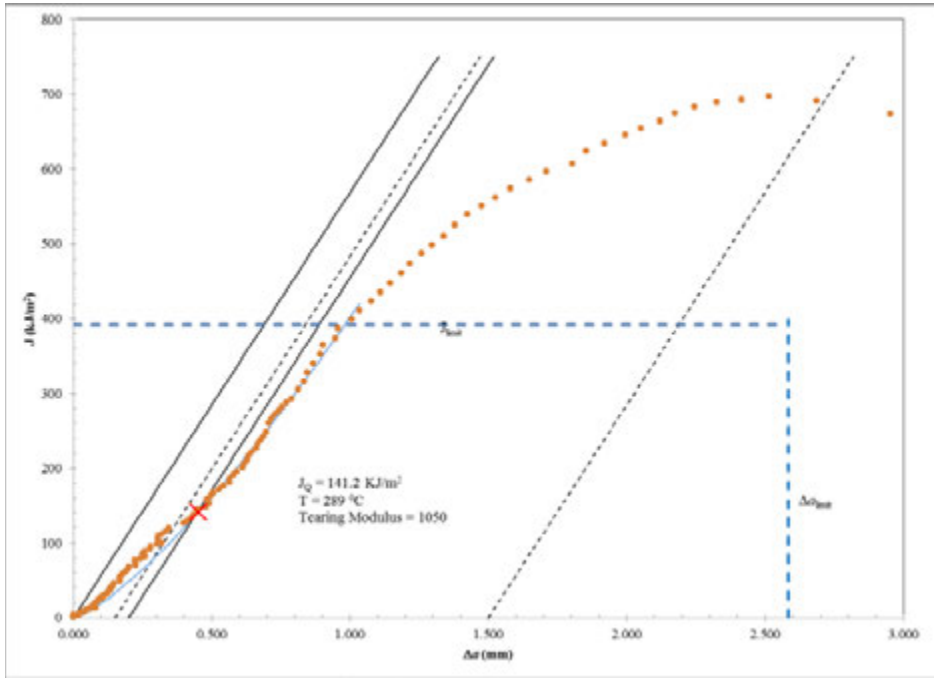

INL precision measurement device

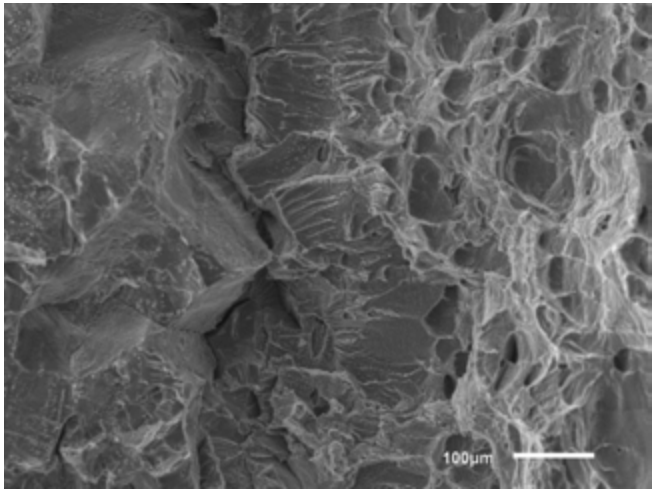


# Annual NSUF Industry Advisory Committee meeting

- **~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 January-February 2017 (coincident with EPRI NPC?)**

# NRC results




Fractograph showing SCC crack (left side), small transgranular transition region and ductile morphology (right side)

Environment: BWR NWC

$a_0$/W ~= 0.491

$\dot{\varepsilon}$ = 0.43 µm/s (1.7E-05 in/s)

$J_Q$* = 141.2 KJ/m$^2$ **

*Not qualified as $J_{IC}$ because of extent of tunneling

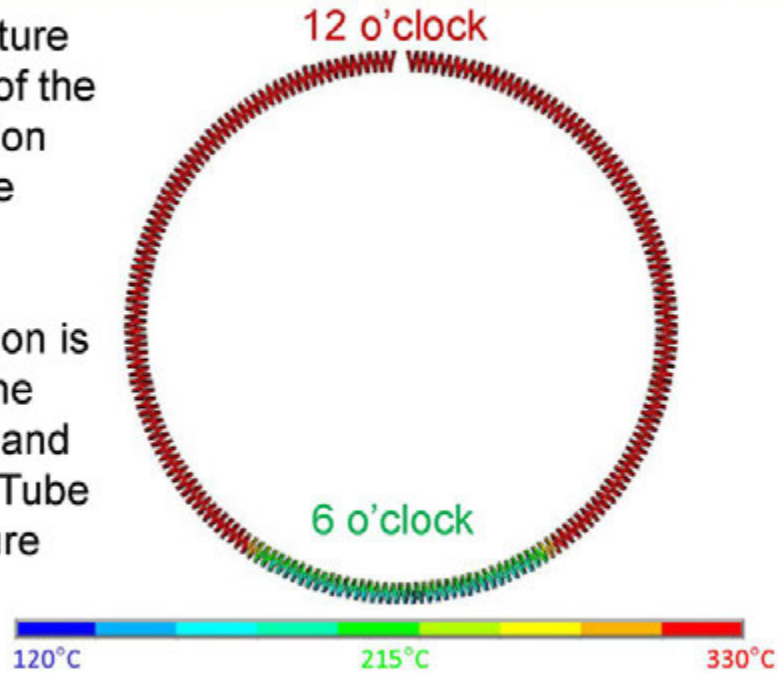
** Toughness could easily range up to ~300; J-R curve follows slope of blunting line to $J_{limit}$.

# AECL project details



## Operating Temperatures

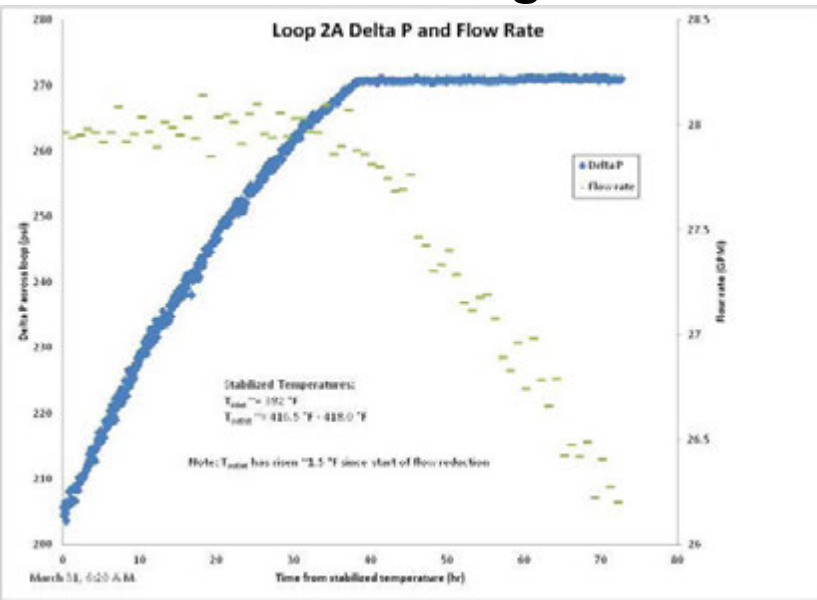
- Irradiation temperature varies as a function of the circumferential location (clock position) of the garter spring on the Pressure Tube.
- The 6 o'clock position is "pinched" between the "hot" Pressure Tube and the "cold" Calandria Tube creating a temperature gradient in the component.

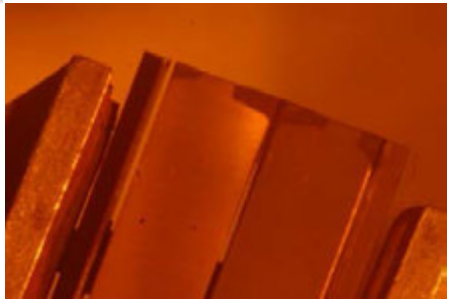


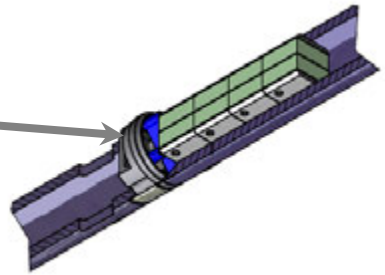

AECL EACL

# Loop 2A flow restriction issue

- **Evolving flow restriction during 1st experiment (EPRI 2)**
  - Flow reduction ~1 GPM/day
  - Water samples from loop contain relatively “high” levels of iron (~ppm)
  - Scrapings from specimens confirm relatively high levels of iron
  - Oxide stripping?
- **Possible re-design of irradiation capsule to increase flow**

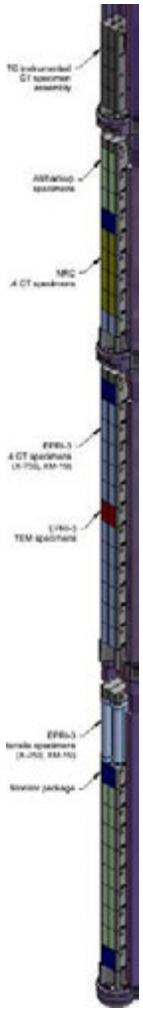


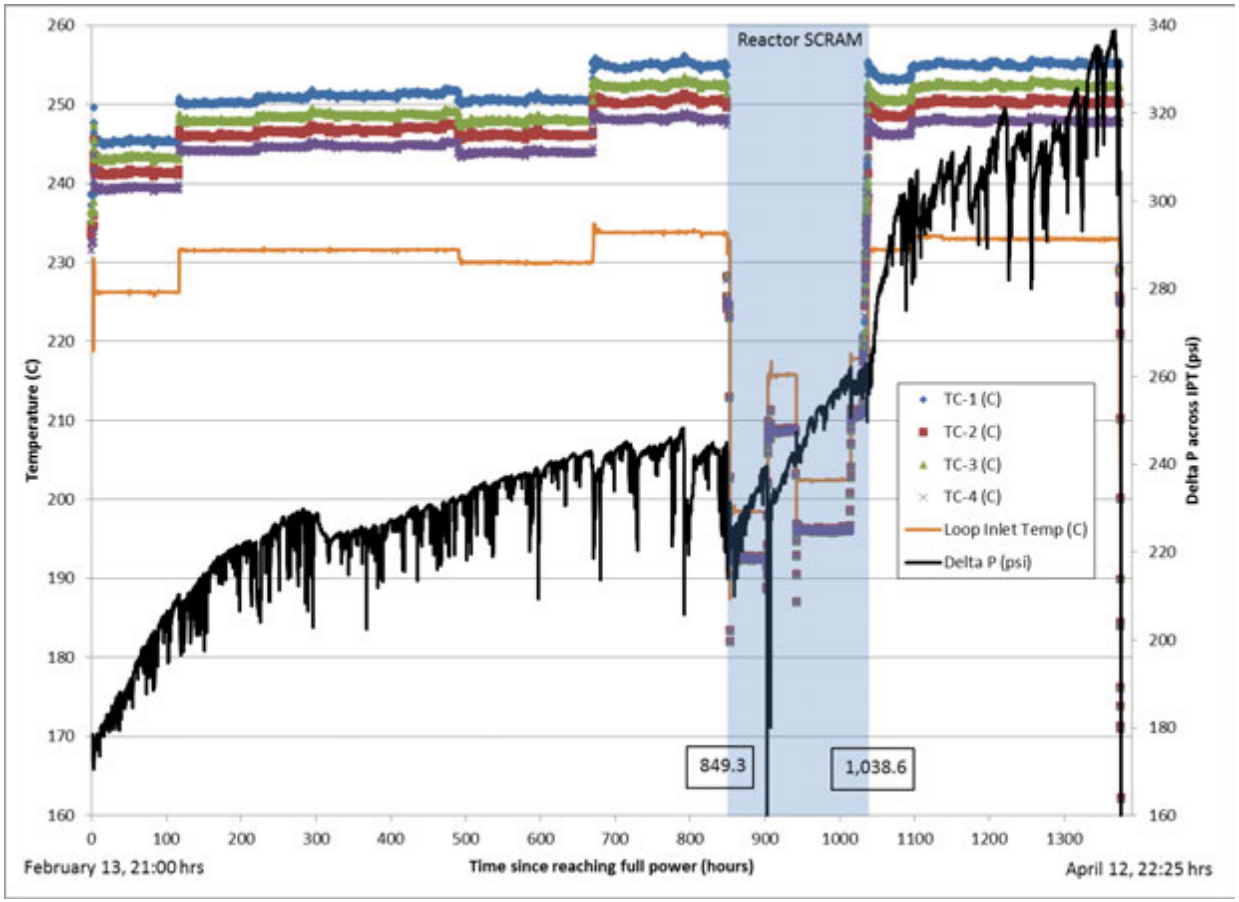





ΔP decreases ~32% with modification

# Loop 2A flow restriction



## EPRI 3 irradiation, cycle 155B

- Flow restriction evolution is much slower but still exists