



Irradiated Materials Testing Programs

Materials Reliability Program

Jean Smith

Sr. Technical Leader

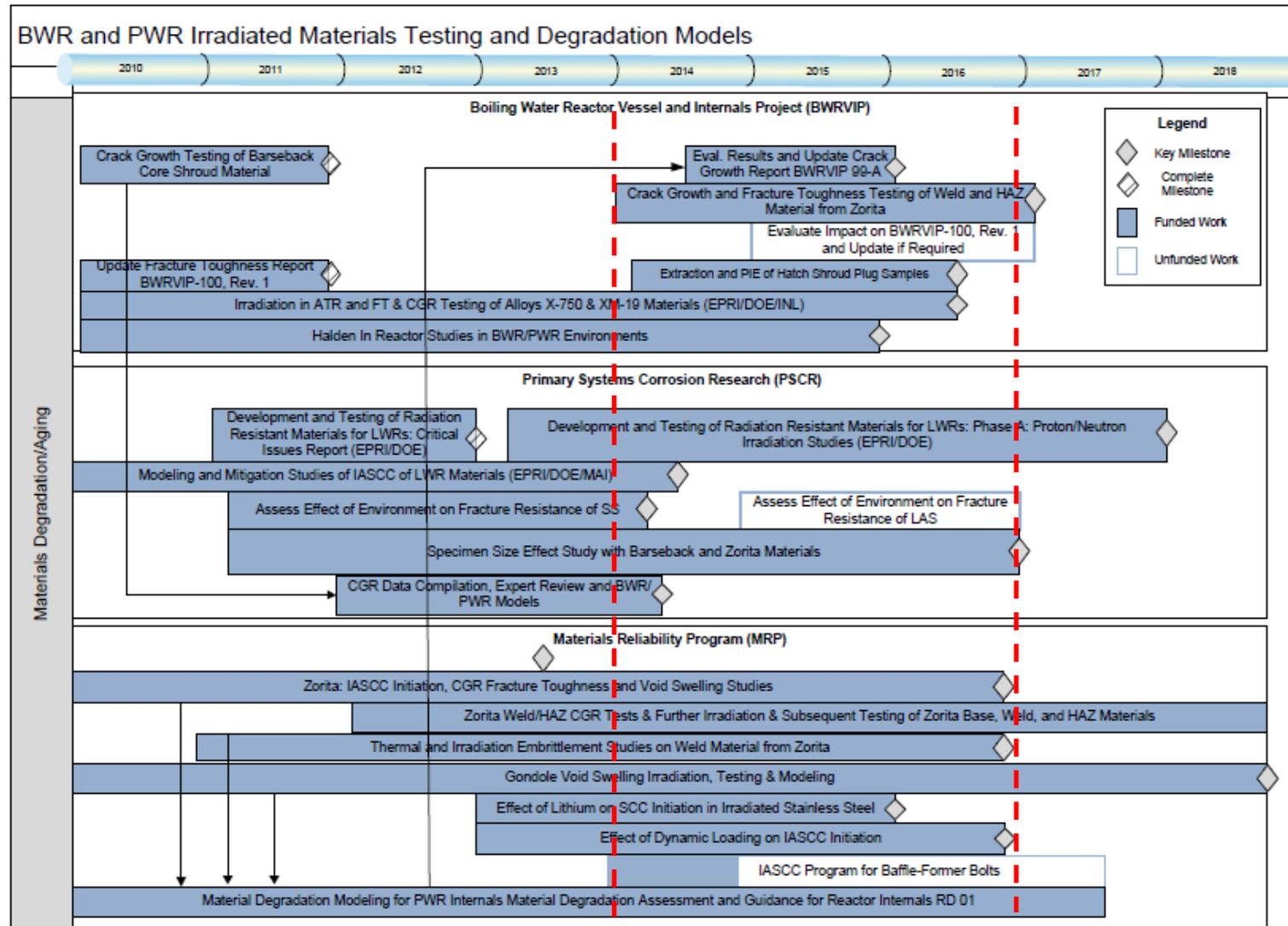
EPRI – NRC Research Materials Meeting

June 3-5, 2014

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EPRI Irradiated Materials Testing and Degradation Models Roadmap





Zorita Internals Research Project

Zorita Internals Research Project (ZIRP)

Objective: Characterize the effects of neutron irradiation on the mechanical and microscopic properties of stainless steel materials irradiated under service conditions to increase the understanding of fluence effects at end of service life (40, 60 years and beyond)



Jose Cabrera NPP “Zorita”
Westinghouse design
1968 – 2006 (~26 EFPY)

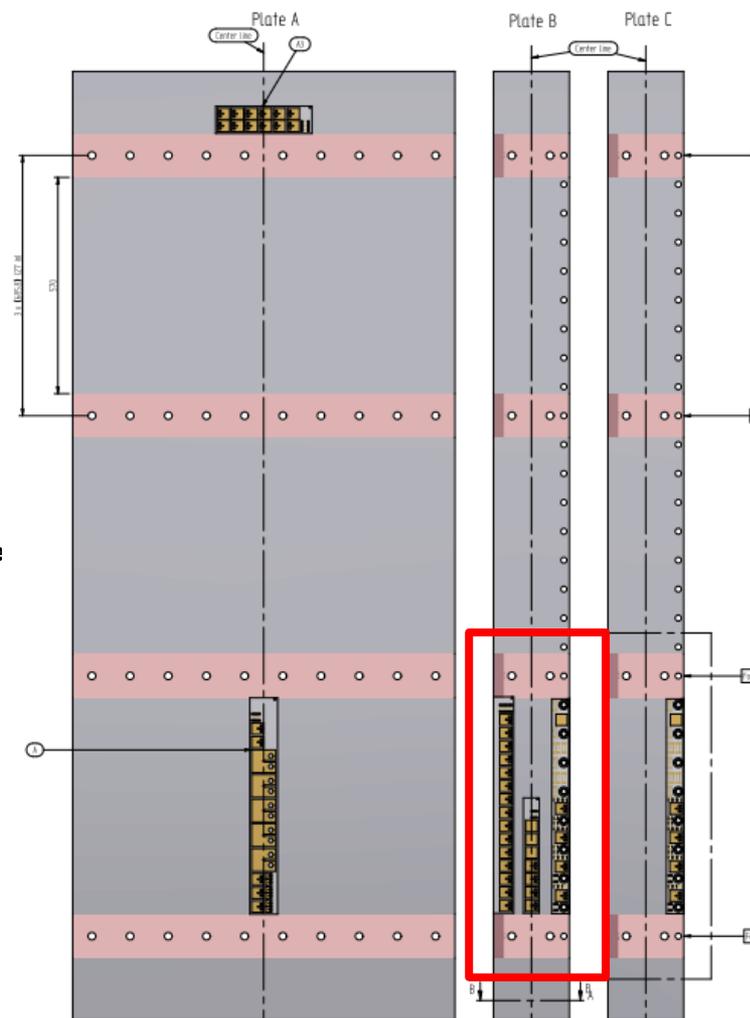
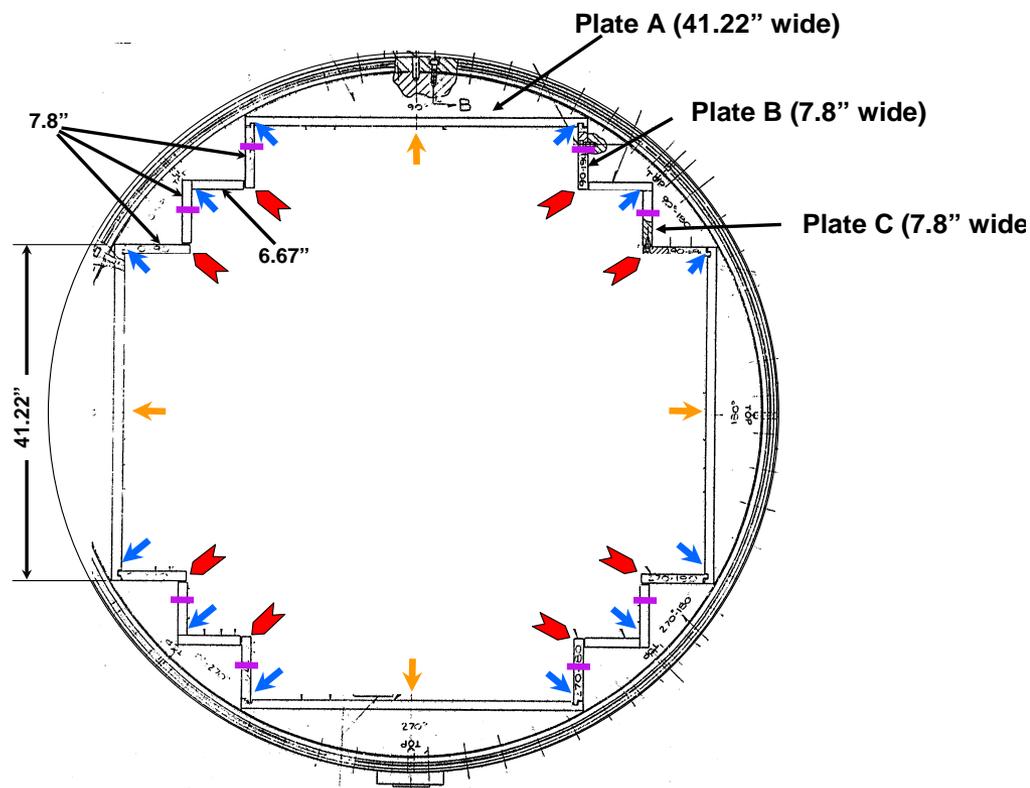
Current Participants

- EPRI
- U.S. NRC
- CSN (Spanish regulator)
- SSM (Swedish regulator)
- Tractebel
- AXPO
- Additional in-kind contribution from Japanese utilities/MHI

Zorita Internals Research Project

Cutting Plan – Baffle Plates

- Baffle plate material between 3rd and 4th formers
- Type 304 Stainless Steel
- Doses ranging from a few dpa to ~50 dpa
- Thickness 28.6 mm
- **ZIRP uses Pieces B1, B2, and B3**



Zorita Internals Research Project

Zorita Materials

B3



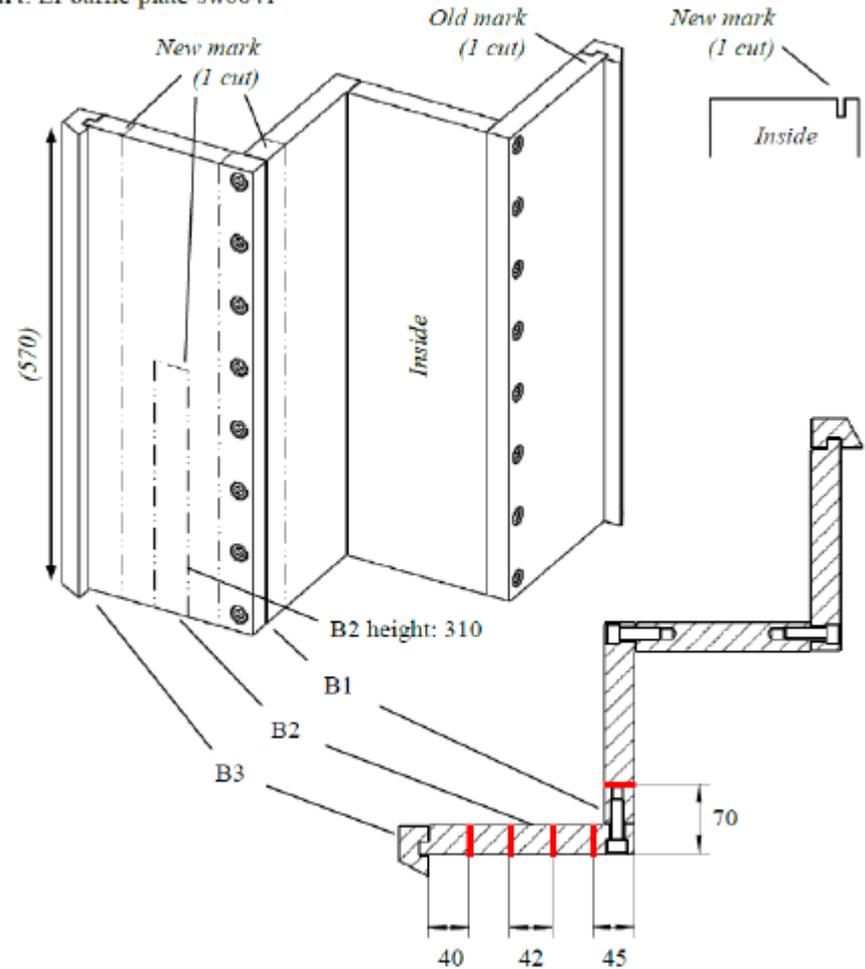
B2



B1



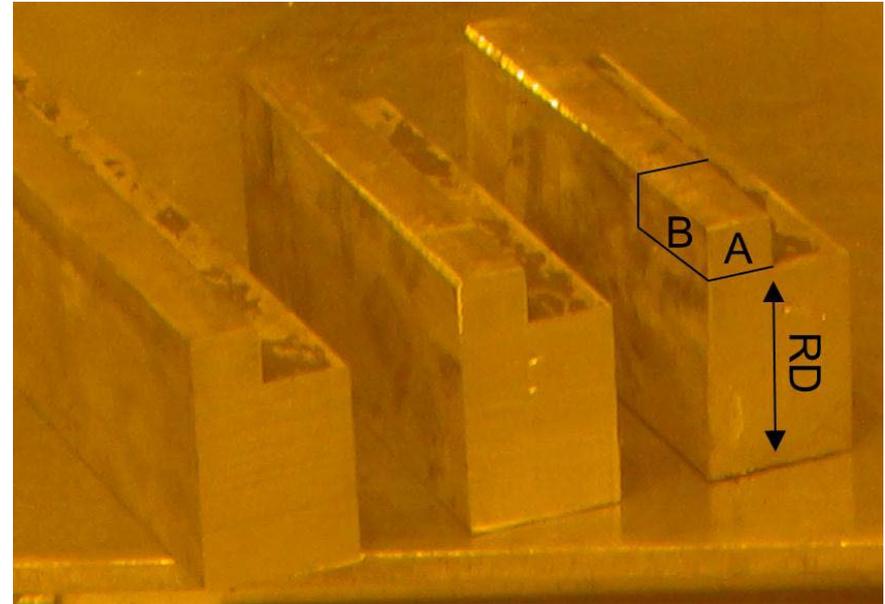
Part: LI-baffle-plate-sw0041



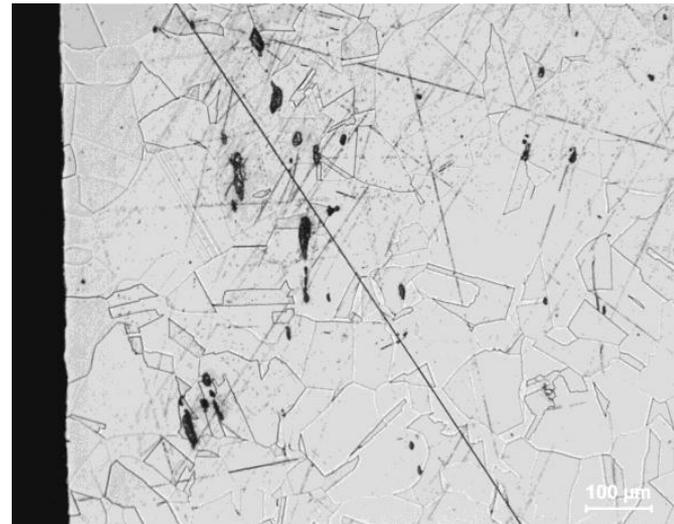
Zorita Internals Research Project

Rolling Direction

Surface A



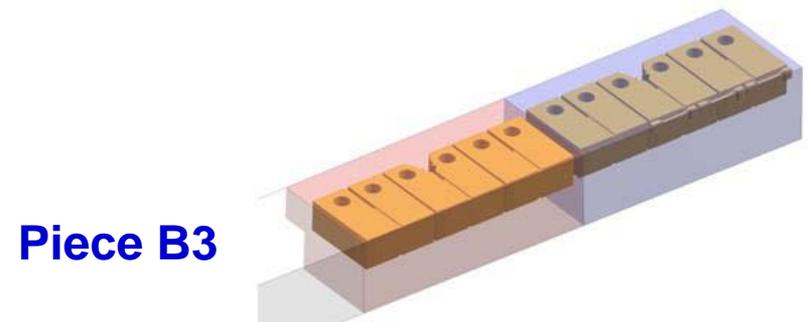
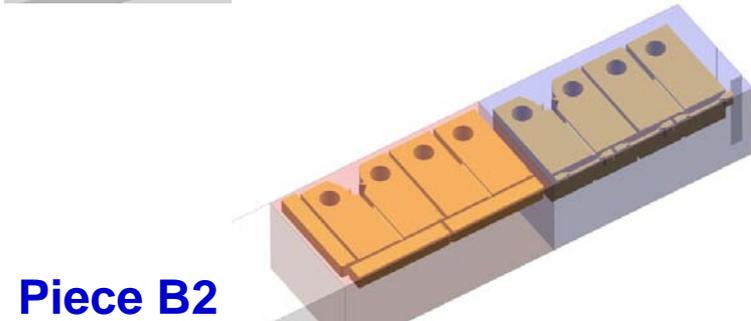
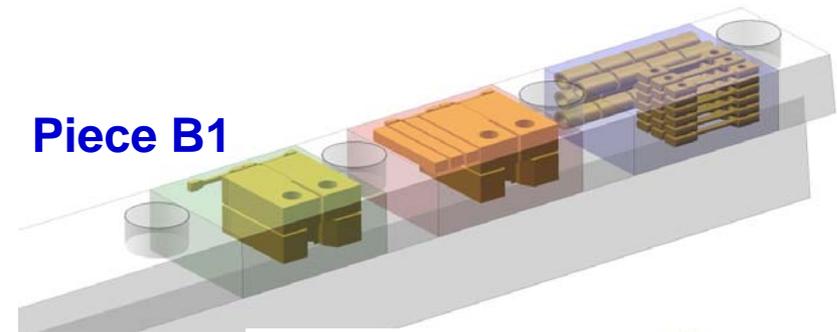
Surface B



Zorita Internals Research Project

Specimen Location – Pieces B1, B2, and B3

- Piece B1 (high dose)
 - Specimens for CGR testing are placed in the top layer closest to the surface that faced the core (highest dose)
 - FT specimens are in the lower layer (lower dose)
 - Location for crack initiation specimens subject to change
- Piece B2 (medium dose)
 - All CT specimens will be machined from the top layer where the dose is highest
- Piece B3 (low dose)
 - CT specimens will be extracted from approximately mid-thickness where the dose is ~10 dpa

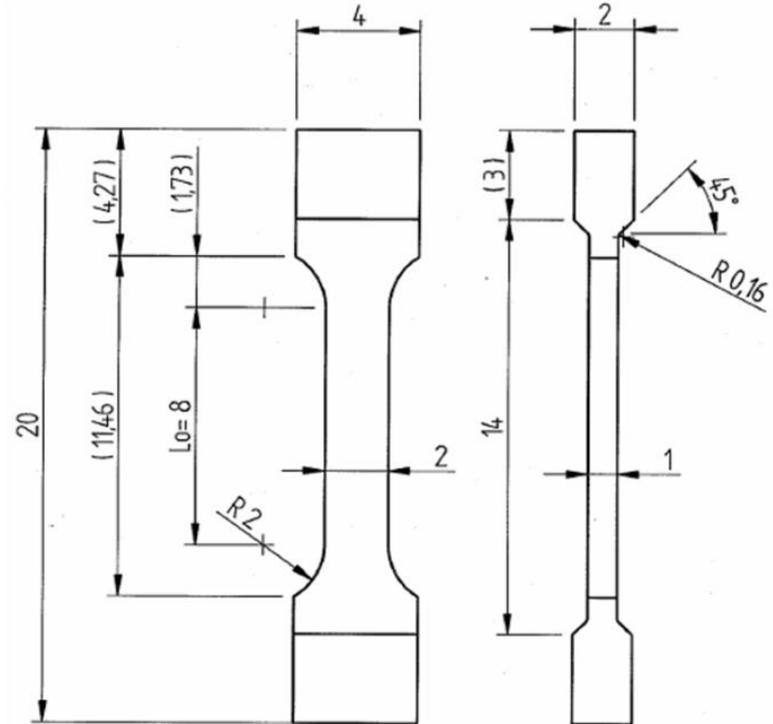


Zorita Internals Research Project

Tensile Testing – Test Matrix & Specimen Geometry

Tensile Testing: **Complete**

Number of Specimens	Dose (dpa)	Test Temperature (°C)
1	>50	RT
2	>50	320
1	~30	RT
2	~30	320
1	~10	RT
2	~10	320



Zorita Internals Research Project

Crack Growth Rate (CGR) Testing

- Testing of different K levels will give information on the K dependency
- Increased K for the first specimens of each dose to 16 & 25 MPa√m
 - K = 11 MPa√m is a relatively low value and may result in very low CGR (and long exposure times)
 - K = 20 MPa√m can be increased to 25-30 MPa√m and still be valid
- Test protocol for the next specimens will be determined on the results from the first test

Specimen Number	Dose Dpa	Temperatures °C	K _I MPa√m
1	~10	290, 320, 340	16 & 25
2	~10	290, 320, 340	TBD
3	~30	290, 320, 340	16 & 25
4	~30	290, 320, 340	TBD
5	>50	290, 320, 340	16 & 25
6	>50	290, 320, 340	TBD

Duration	Autoclave 1	Autoclave 2
April – July 2014	Specimen #3 (30 dpa)	Specimen #5 (50 dpa)
Sept. – Dec. 2014	Specimen #1 (10 dpa)	Specimen #4 (30 dpa)
Feb. – May 2015	Specimen #6 (50 dpa)	Specimen #2 (10 dpa)

Zorita Internals Research Project Fracture Toughness – Test Matrix

Specimen #	Dose (dpa)	Temperature °C	Environment
1	~10	320	Air
2	~10	320	Air
3	~10	320	Primary Coolant
4	~10	320	Primary Coolant
5	~30	320	Air
6	~30	320	Air
7	>50	320	Air
8	>50	320	Air

- Fracture Toughness Testing: September/October 2014

Zorita Internals Research Project

Crack Initiation Testing

- Testing to begin in 4Q2014
- Tests are scheduled to last 9,000 hours

Stress Level % of YS_{irr}	Type of Specimen	No. of Specimens
100	O-ring	2
80	O-ring	2
70	O-ring	2
60	O-ring	3
50	O-ring	3
40	O-ring	2
25	O-ring	2
100	UCL	3
80	UCL	3
70	UCL	3
60	UCL	3
50	UCL	3
40	UCL	3

320 °C, 1000 ppm B, 2 ppm Li, DH 30 cc/kg

Zorita Internals Research Project

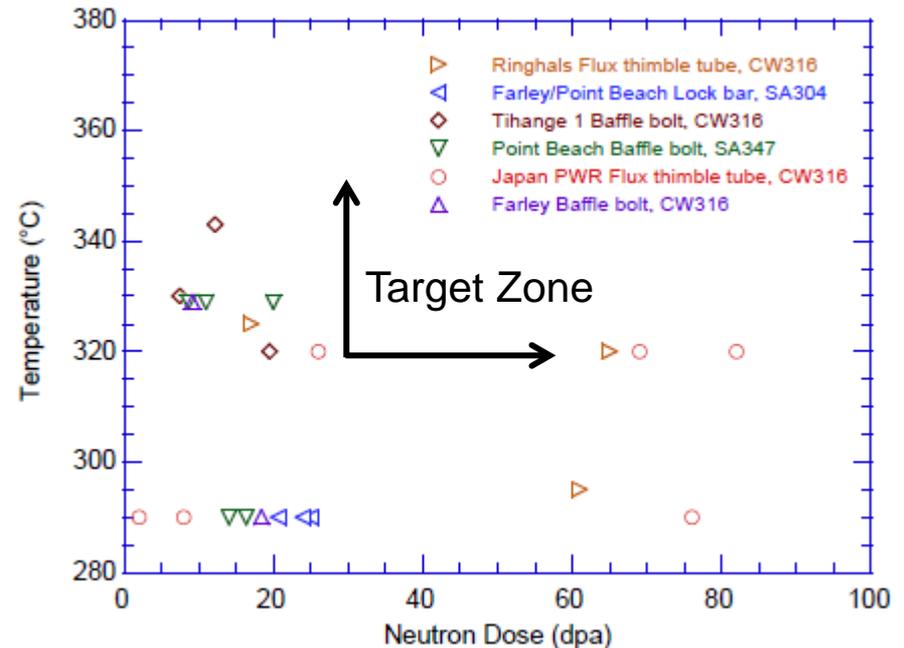
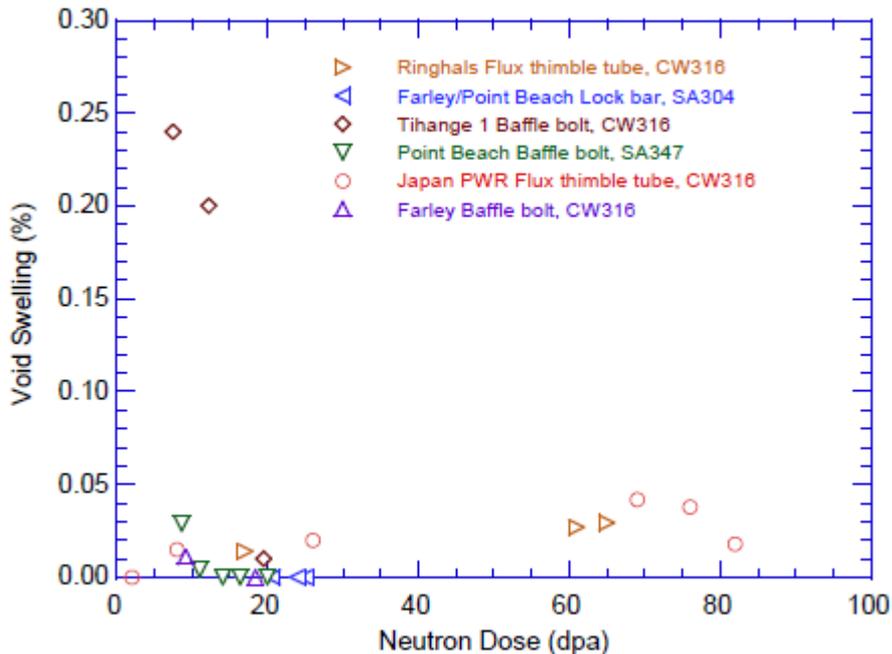
Microstructural Characterizations

- Six (6) specimen blanks each 5x5x30mm
 - Optical microscopy
 - Grain boundary characterization
 - Radiation-induced segregation
 - Hardness measurements
 - TEM observations
 - Frank loop size and density
 - Void size and density
 - Gas analysis
 - Helium and hydrogen

Zorita Internals Research Project

TEM Specimen Considerations

- Example of field data on swelling from NUREG/CR-7027
- Swelling more dependent on temperature than dose
- Insignificant swelling on 304 SS irradiated about 290 °C and about 20 dpa



Zorita Internals Research Project

Proposed TEM Specimen Matrix

Sample Number	Material	Irradiation Fluence	Irradiation Temperature
1	304 SS (Baffle plate)	ϕ_1 (e.g. 30dpa)	T_1
2			(e.g. 320°C)
3		ϕ_1 (e.g. 30dpa)	T_2
4			(e.g. 350°C)
5		ϕ_2 (e.g. 50dpa)	T_1
6			(e.g. 320°C)

The diagram shows two curved arrows on the right side of the table. The top arrow, labeled 'Temp. dependence', spans rows 1 and 2, and rows 3 and 4, indicating that temperature is varied within each fluence group. The bottom arrow, labeled 'phi dependence', spans rows 1 and 3, and rows 5 and 6, indicating that fluence is varied within each temperature group.

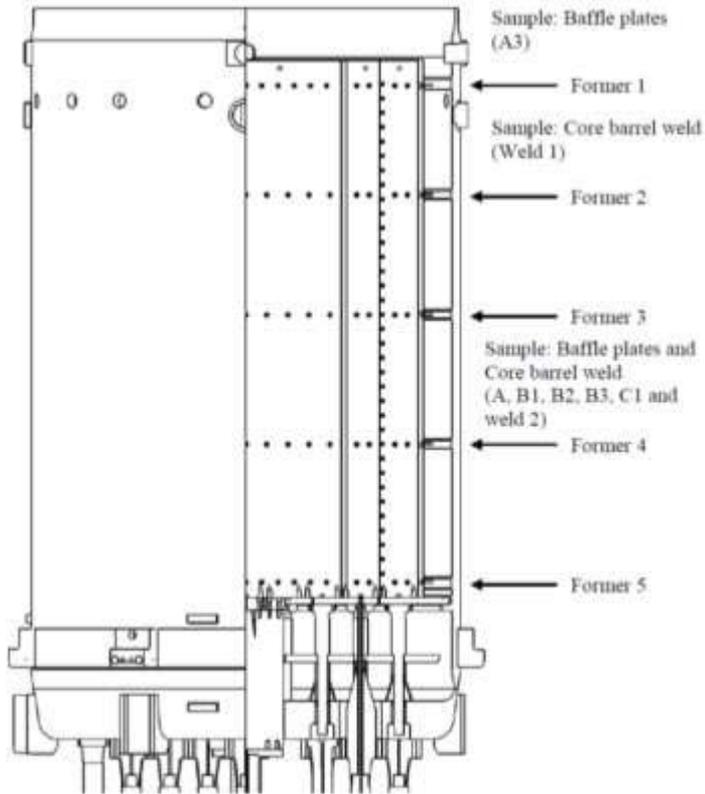
- ❖ $T_2 > T_1$ (Ideally, T_1 is almost 320°C or higher)
- ❖ $\phi_2 > \phi_1$ (Ideally, ϕ_2 is as high as possible)



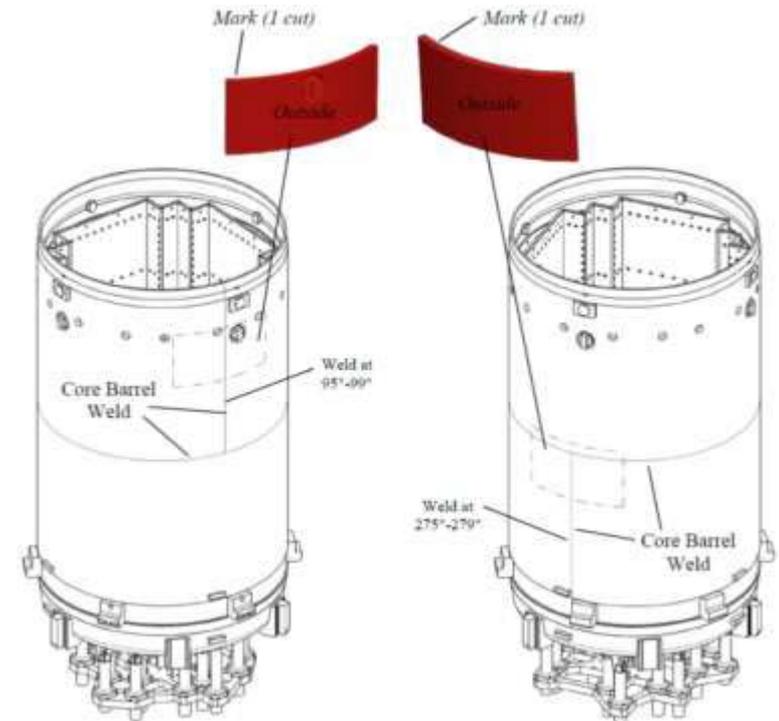
Other Zorita Materials Projects

Testing of Zorita Weld Materials

Weld Locations



Vertical Locations of Weld 1 and Weld 2 in the Internals Assembly



Locations on the core barrel from which Weld 1 (left) and Weld 2 (right) were extracted

Testing of Zorita Weld Materials

Weld Piece 2



Testing of Zorita Weld Materials

Thermal and Irradiation Embrittlement and Environmental Effects Testing of Stainless Steel Welds

- Determination of the combined effects of irradiation and exposure to elevated temperature on embrittlement of stainless steel welds and characterization of environmental effect on fracture toughness in irradiated stainless steel welds
- Gap P-AS-13 (high): Thermal & Irradiation Embrittlement Synergistic Effects on CASS and Stainless Steel Welds

Specimen size	Orientation	# of specimens	Dose dpa	Environment	Temperature °C
0.5TCT	T-S	2	~2	Air	RT
0.5TCT	T-S	2	~2	Air	320
0.5TCT	T-S	2	~2	PWR	150 ¹⁾
0.5TCT	T-S	2	~2	PWR	320

1) Simulated shutdown conditions to be defined in terms of temperature and water chemistry (B, Li, dissolved hydrogen, presence of oxidizing species)

Testing of Zorita Weld Materials

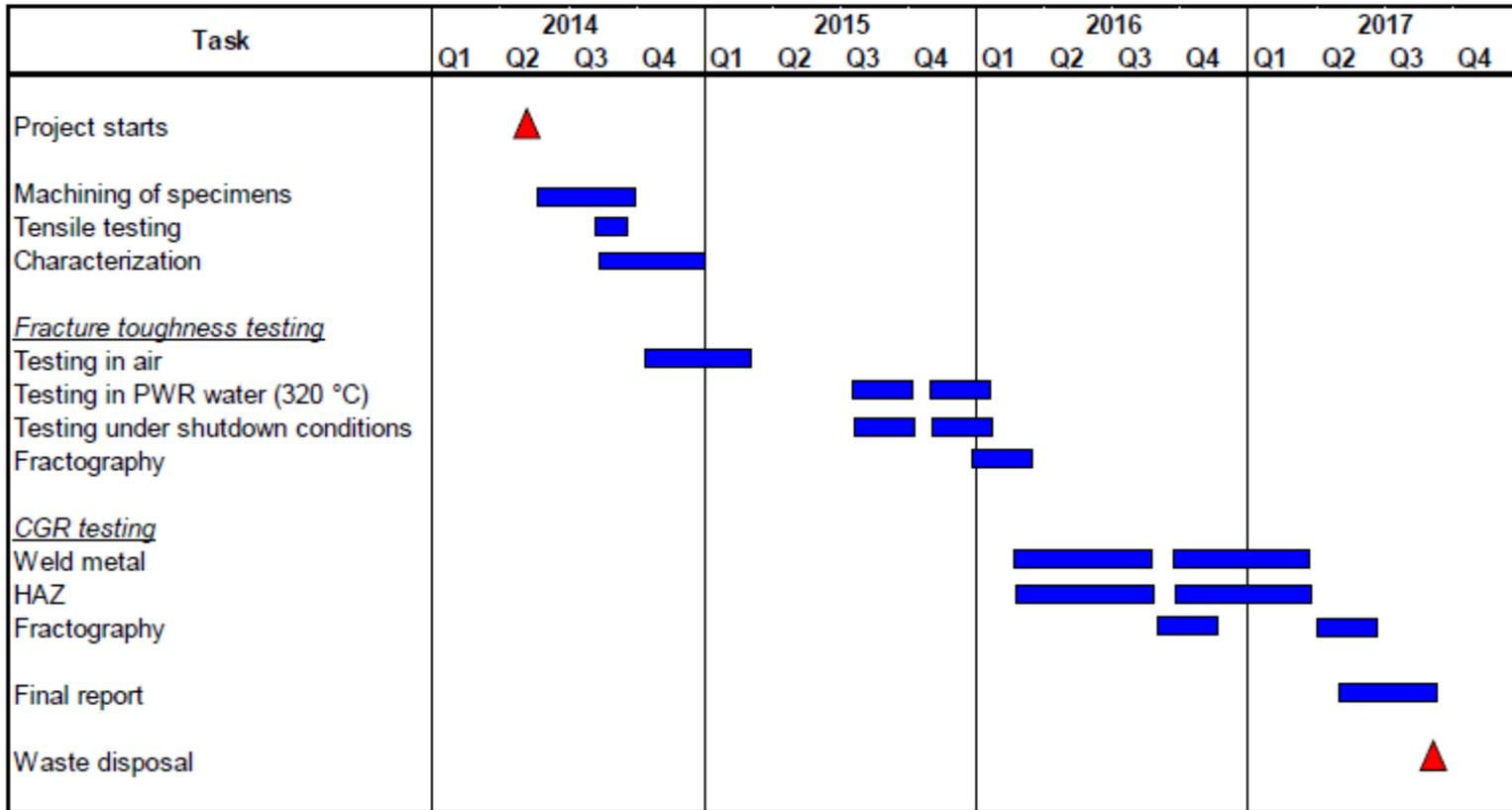
CGR Testing of Irradiated SS Weld and HAZ Materials

- Generation of IASCC CGR data in irradiated stainless steel weld and HAZ materials for comparison to existing data for base materials
- Gap P-AS-14a (high): IASCC Characterization: Generic Data Needs

Specimen	Material	Orientation	Test Temperatures °C	$K_I^{1)}$ MPa \sqrt{m}
1	Weld	T-S	290, 320, 340	~10 & 20
2	Weld	T-S	290, 320, 340	~15 & 25
3	HAZ	~L-S or ~T-S	290, 320, 340	~10 & 15
4	HAZ	~L-S or ~T-S	290, 320, 340	~20 & 25

1) The highest K level will be determined by the yield strength of the material, taking into account K validity concerns for irradiated materials.

Testing of Zorita Weld Materials Estimated Schedule



Determination of IASCC CGR, Initiation Rate, and Void Swelling in Zorita Material After Post-Reactor Irradiation

- *Objective*

- Evaluation of IASCC crack initiation and crack growth rates and degree of void swelling in highly-irradiated (near end-of-life conditions) stainless steel base metal and welds

- *Materials*

- Baffle plate material and core barrel weldment samples from Zorita

- *Testing*

- Materials will be exposed to prototypical PWR conditions (temperature, neutron spectrum and flux) for an additional exposure of ~15 dpa (or up to 30 dpa)
- IASCC crack initiation and growth rates and degree of void swelling using testing protocols similar to those implemented for the Zorita

- *Gaps*

- P-AS-14a (high): IASCC Characterization: Generic Data Needs
- P-AS-15 (medium): Void Swelling of Stainless Steels

Additional Irradiation & Testing RFP Categories

Table 1-1 Specimens in the Base Irradiation Category

Purpose	Specimen Type	Total Number of Specimens	Additional Accumulated Dose (dpa)
Crack Growth Rate (CGR)	0.5T CT	3	15
Crack Initiation (CI)	O-R / UCL	3	15
Yield strength (YM)	Tensile	3	15
Void Swelling (VS)	TEM	3	15

Table 1-2 Specimens in the Extended Irradiation Category

Purpose	Specimen Type	Total Number of Specimens	Additional Accumulated Dose (dpa)
Crack Growth Rate (CGR)	0.5T CT	9	15
Crack Initiation (CI)	O-R / UCL	6	15
Yield strength (YM)	Tensile	9	15
Void Swelling (VS)	TEM	3	15

Table 1-3 Specimens in the Extended including High Dose Category

Purpose	Specimen Type	Total Number of Specimens	Additional Accumulated Dose (dpa)
Crack Growth Rate (CGR)	0.5T CT	9	15
		3	30
Crack Initiation (CI)	O-R / UCL	6	15
		--	--
Yield strength (YM)	Tensile	9	15
		3	30
Void Swelling (VS)	TEM	3	15
		3	30

Additional Irradiation & Testing

Next Steps

- Issue RAIs to potential contractors
 - Common set of questions plus proposal-specific questions
- Explore sources of additional funding



Effect of Lithium on IASCC Initiation

MRP: Effect of Lithium on IASCC Initiation

Objective

- Determine the effect of lithium (Li) on the rate of IASCC initiation for comparison to recent data generated by EDF suggesting increased Li concentration may enhance IASCC initiation rate
- Li levels of 2.0 and 8.0 ppm will be studied at 3 different specimen loading conditions (35, 45, and 60% of yield strength)

EDF Results

- Tests performed on Chooz A corner (Type 304 SA)
- Li content: 3.5 ppm compared to 2.1 ppm (pH value was similar for both Li levels)
- Time for cracking decreased when Li content increased
- Cracking observed at lower applied strain with Li=3.5 ppm than with Li = 2.1 ppm

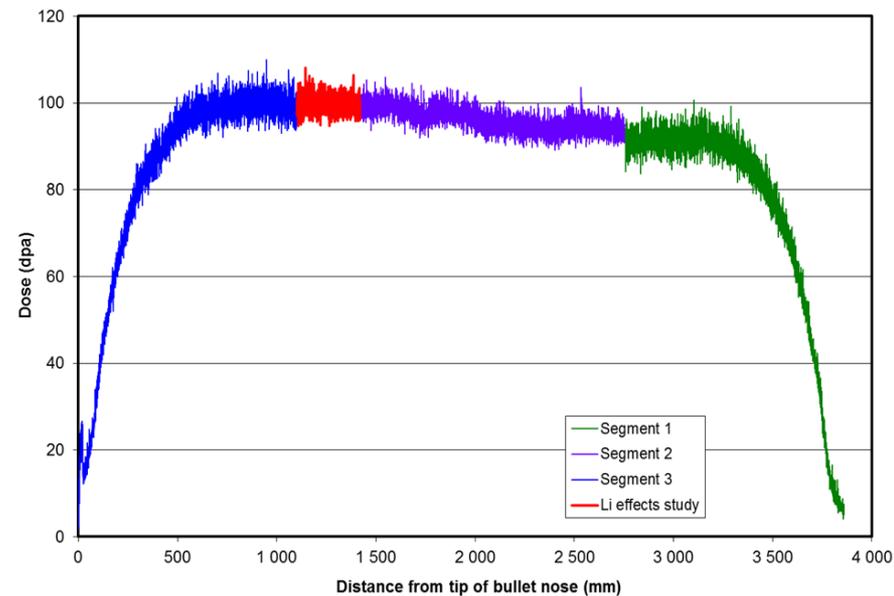
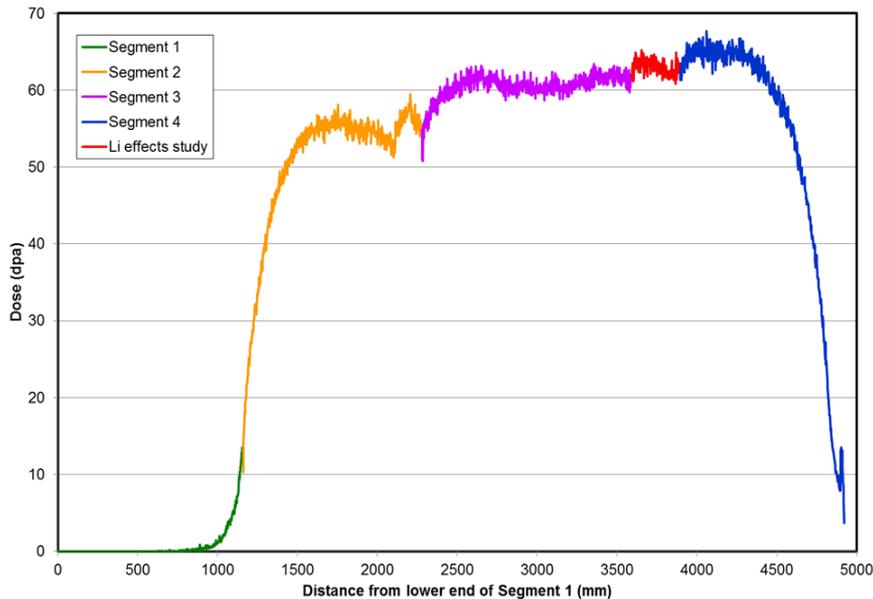
IMT Gap

- **P-AS-14a (high):** IASCC Characterization: Generic Data Needs

MRP: Effect of Lithium on IASCC Initiation

Flux thimble tubes from Ringhals 2: maximum doses of 65 and ~100 dpa

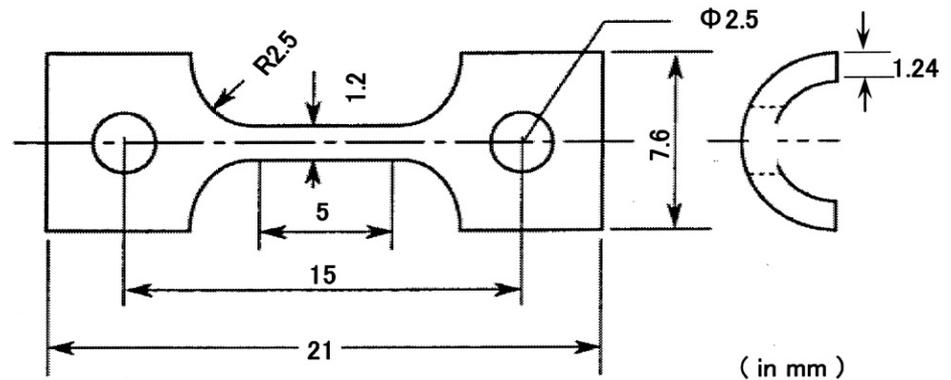
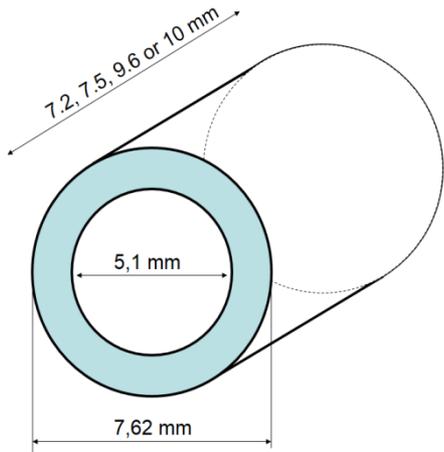
Specimens for this project were machined from tube pieces ~1 m from the bullet nose



MRP: Effect of Lithium on IASCC Initiation

O-ring and UCL specimens

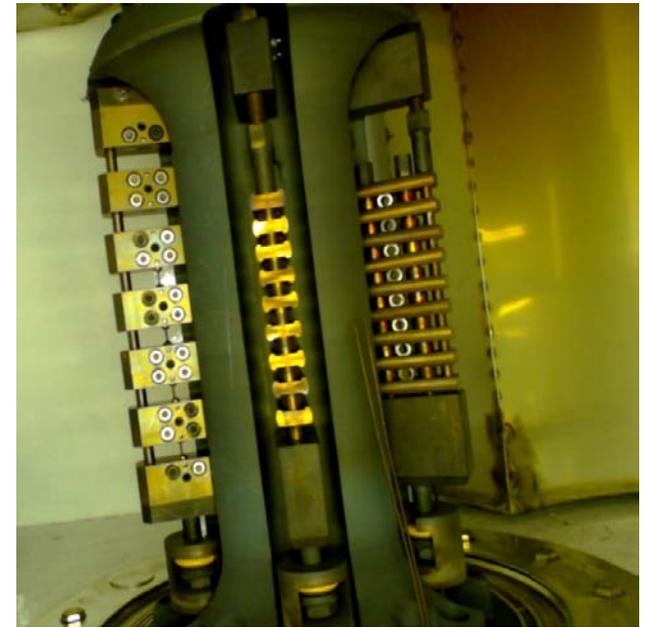
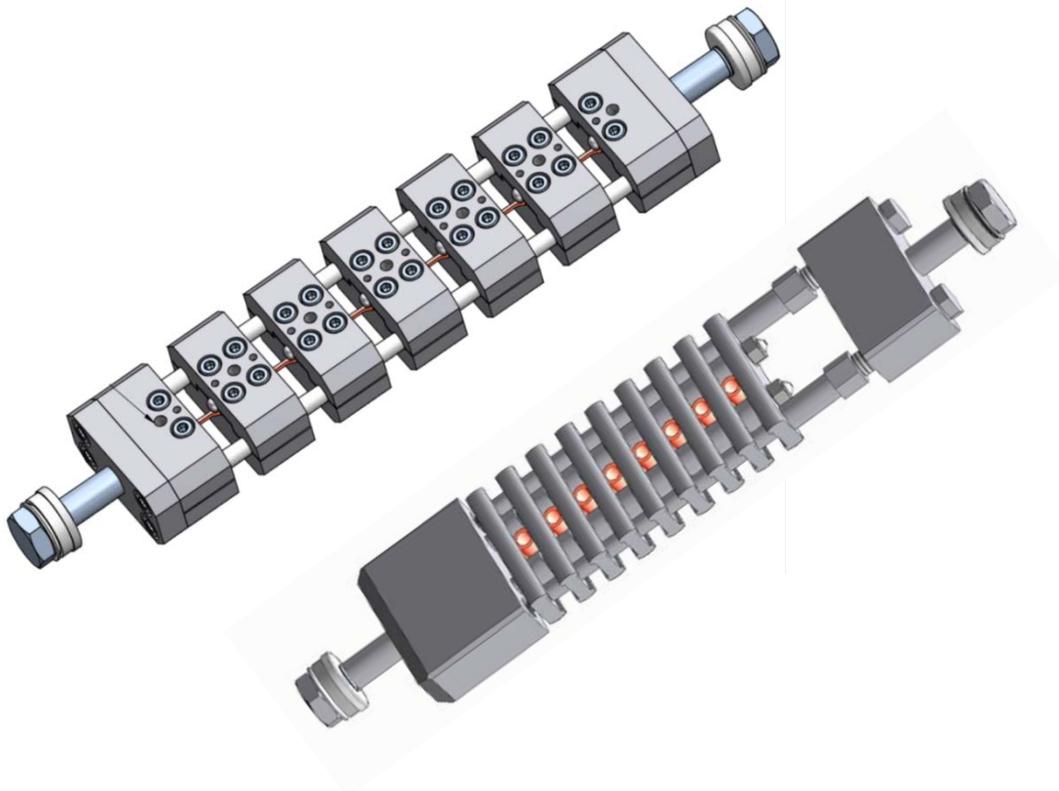
- FE modeling to determine loads for the O-rings
- UCL specimens similar to ZIRP



MRP: Effect of Lithium on IASCC Initiation

One autoclave with 5 load trains

- Up to 6 UCL specimens per load train
- Up to 8 O-ring specimens per load train



MRP: Effect of Lithium on SCC Initiation

Test Matrix

- 5000 hours of exposure per test, or less if all specimens have failed
 - Increase load after ~3,000 hours if no failures
 - Test 1: Loads increased to 70, 52.5, and 40% after 3,000 hours
- Visual inspection of all specimens tested, including possible non-failed specimens
- Fractographic examination by scanning electron microscopy (SEM) of selected failed specimen to determine the failure mode
- High-resolution microscopy (FEGSEM and TEM) on selected samples
 - Study the influence of Li on the oxides formed on the sample surfaces
 - Improve knowledge of initiation of IASCC in SS at high dose
- Determine time to failure as functions of Li content, dose, and stress

Dose (dpa)	Stress Level (% YS _{irr})	Specimen Type	No. of Specimens
60	60	O-ring	2
60	45	O-ring	2
60	35	O-ring	2
100	60	O-ring	2
100	45	O-ring	2
100	35	O-ring	2
60	60	UCL	2
60	35	UCL	2
100	60	UCL	2
100	35	UCL	3

Test matrix repeated in each environment

1: Boron tbd, 2.0 ppm Li, 30 cc/kg H₂, T=340 °C, pH_{300°C} = 7.2

2: Boron tbd, 8.0 ppm Li, 30 cc/kg H₂, T=340 °C, pH_{300°C} = 7.2

MRP: Effect of Lithium on IASCC Test Schedule

Task Description	Completion Date
Machining of Test Specimens	June 2013
Crack Initiation Testing	November 2014
Visual Inspection and Fractography	March 2015
Draft Final Report	April 2015
Final Report	June 2015



Gondole Void Swelling Project

Gondole Void Swelling Project

Objective: Characterize the development of void swelling in stainless steels under PWR conditions (temperature, spectrum, flux) over time as a function of neutron fluence

- Virgin PWR internals materials & pre-irradiated materials (Types 304 & 316 SS)
 - Phase 1 (2004-2010): ~14 dpa on virgin materials and up to 85 dpa on pre-irradiated materials
 - Phase 2 (2011-2015): Additional 15 dpa
- Gondole Test Conditions
 - CEA Osiris mixed spectrum reactor
 - 5 cycles of irradiation for total of ~15 dpa
 - 360°C, fast neutron flux $2 \times 10^{14} \text{ n} \cdot \text{cm}^{-2} \cdot \text{s}^{-1}$ ($E > 1 \text{ MeV}$)



Gondole Void Swelling Project

- Measurements
 - Density measurements (5 each after each phase)
 - Profilometry after each phase
- Characterizations
 - Microstructural evaluation by TEM to be compared to initial state
 - After Irradiation Phase 8: examination of 5 TEM thin foils machined from discs or taken from density samples
 - Measurement of gas content on 5 samples after Irradiation Phases 5 and 8
- Modeling
 - Empirical and quantitative modeling of results

Gondole Void Swelling Project

- Total cumulated dose at the end of Phase 6 approx. 19-20 dpa
- Currently in irradiation Phase 7
 - Began 10 cycles in January 2013
 - Ends June 2014
 - Goal 5.1 dpa (iron)
- Irradiation Phase 8
 - 10 cycles beginning November 2014
 - Goal 5.1 dpa (iron)



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