Potassium Hydroxide (KOH) Qualification Program

Overview

Keith Fruzzetti, PhD
Senior Technical Executive

EPRI-NRC Research Discussion on KOH Qualification

Webcast
20 October 2017

Other EPRI Technical Team Members

Peter Chou
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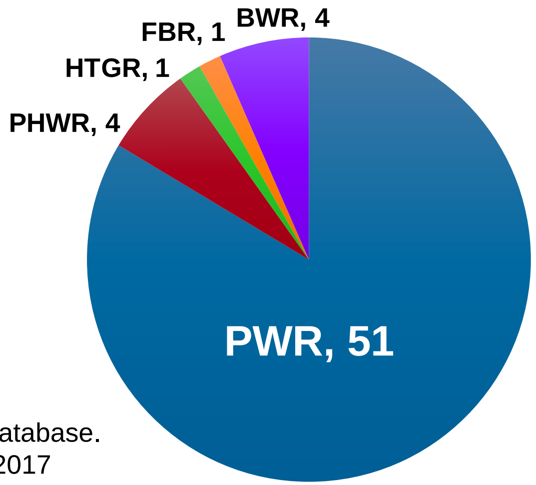
Why Investigate KOH?

- Some utilities challenged / unable to procure Li-7 in 2015
  - Production only in China and Russia
  - Cost has increased significantly since 2015
  - Dependability of continued supply is uncertain
- Operational considerations
  - Flexible power operations will GREATLY increase Li-7 demand
  - Growing PWR fleet will require even more Li-7
- Molten salt reactors would dramatically increase demand
  - A single Molten Salt Reactor (1000 MWe) requires as much Li-7 as ~750 commercial PWR units (annualized basis)

GAO-13-716, "Managing Critical Isotopes: Stewardship of Lithium-7 Is Needed to Ensure a Stable Supply", Sep. 2013.

Press Release, House Committee on Science, Space, & Technology, "GAO Raises Questions about Adequate Supply of Lithium-7 for Nuclear Power Reactors", Oct 9, 2013.

Reactors Under Construction by Type



Source: IAEA PRIS Database.
Updated 08 Aug 2017

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Additional Drivers for KOH

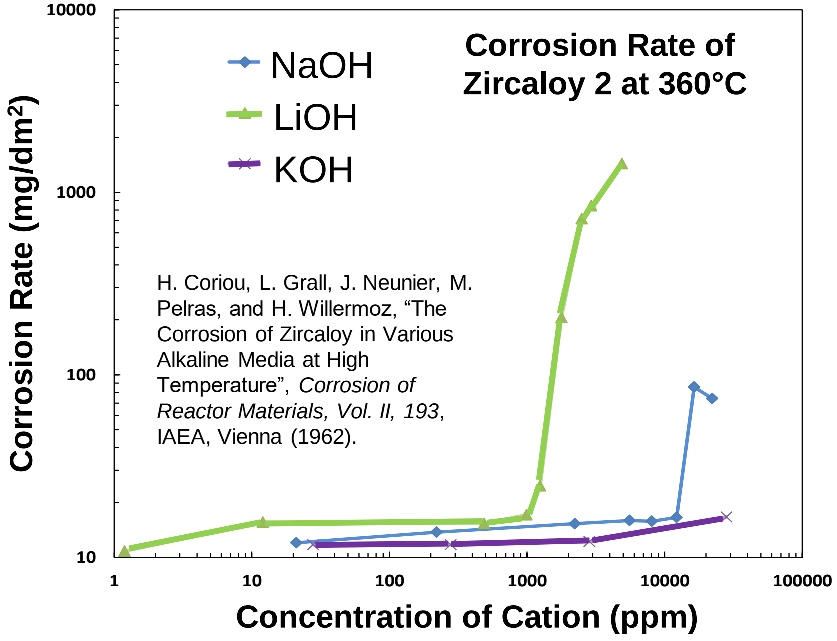
- **Lower Operational Costs**
  - Estimated savings <u>per year</u> (based on median values)

    Each PWR unit: $90k

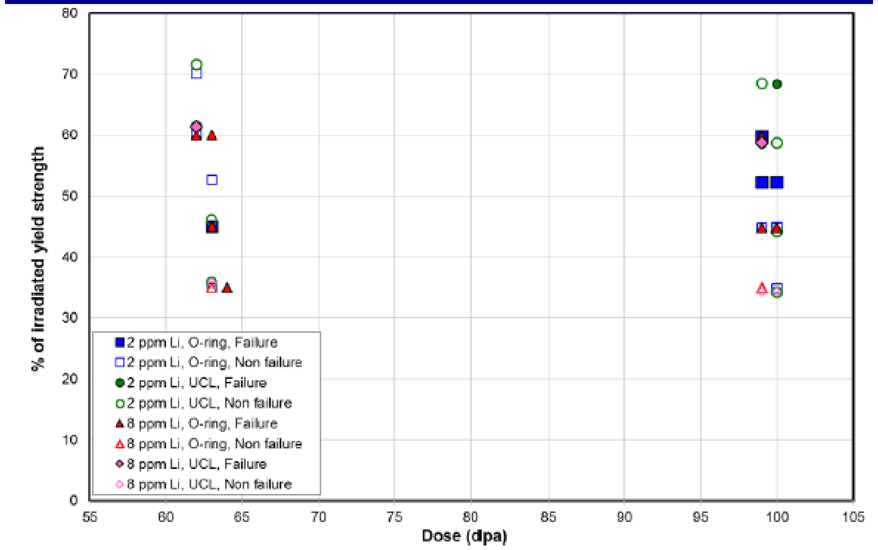
    178 EPRI Member PWRs: $16M

- **May be more beneficial for Fuel**
  - Data indicate much lower corrosion rates
  - Potential CIPS* mitigation strategy

- **May have a beneficial effect on IASCC** initiation**
  - Much lower lithium concentrations possible with KOH



IASCC appears to initiate at shorter times and at lower stress level at 8 ppm Li (vs 2 ppm Li) [MRP-413, Fig 5-1]



*Crud Induced Power Shift, **IASCC: Irradiation Assisted Stress Corrosion Cracking

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KOH Qualification Advisory Committee

- **Objective**: Challenge and Improve Qualification Plan
  - Individual webcasts with each discipline prior to in-person meeting
  - In-person Meeting: April 18-19, 2017
  - Webcast (focused on finalizing materials testing): September 26, 2017
    - Organizations and disciplines represented on the committee

      - AREVA
      - CEZ/UJV Rez
      - Duke
      - Entergy
      - Exelon
      - FENOC
      - Luminant
      - PAKS
      - SNC
      - Vattenfall (Ringhals)
      - Westinghouse

      - Chemistry, Fuels, Materials, Radiation Safety

Detailed Plan for Qualifying KOH Updated (Summary)

Qualification Actions Required Prior to Start of Demonstration

			Schedule
PSCR	Materials Testing	• Initiation & Crack Growth Rate Testing – Non-irradiated testing • Stainless Steel, Alloy 600 – Irradiated testing • Stainless Steel	• 2017-2020
FRP	Fuels Testing and Exams	• Fuel Vendor Assessment • Experimental Loop Testing • Fuel Exams	• 2017 • 2018-2020 • Plant Trial*
RS	Radiation Fields and Radwaste	• Activation species and dose pathways • Effect on plant radiation fields • Effluent and radioactive waste handling	• 2017-2019
Chem	Chemistry/pH Control	• System review and impacts • High temperature chemistry (MULTEQ) • Purity specifications • Multiple alkali (Li & K) modeling and control	• 2016-2019

* Plant Trial to follow Qualification (3 cycles of operation with KOH)

EPRI Fuel Reliability Program KOH Strategy

Literature Review (completed December 2015, unpublished)

- Fuel and chemistry literature reviewed
- Literature show lower corrosion rate for KOH compared to LiOH
- No showstoppers noted for Zr4 (expected to be conservative for Zirlo™ and M5™)

Formal Fuel Vendor Qualification Plans (2017)

- Vendors to provide a formal list of qualification requirements
  - AREVA: Draft report received and under review
  - Westinghouse: Executed contract with Westinghouse, work has begun

Laboratory Experiments (2018 – 2020)

- Details to be finalized based on Fuel Vendor Reviews
- Five different test loops are under consideration
  - Determination made after requirements are fully defined in concert with Fuel Vendors
  - AREVA (Beatrice or CIRENE Loop); Studsvik (Murer or Dissolved Hydrogen Loop); Westinghouse (WALT Loop)

Plant Demonstration (3 cycles)

- Demonstration plan depends on utility, fuel vendor, and plant operations
  - At least two poolside oxide campaigns will be required
    - Crud scrape very likely to be needed
  - Hot cell examinations depend on vendor reviews (may not be required)

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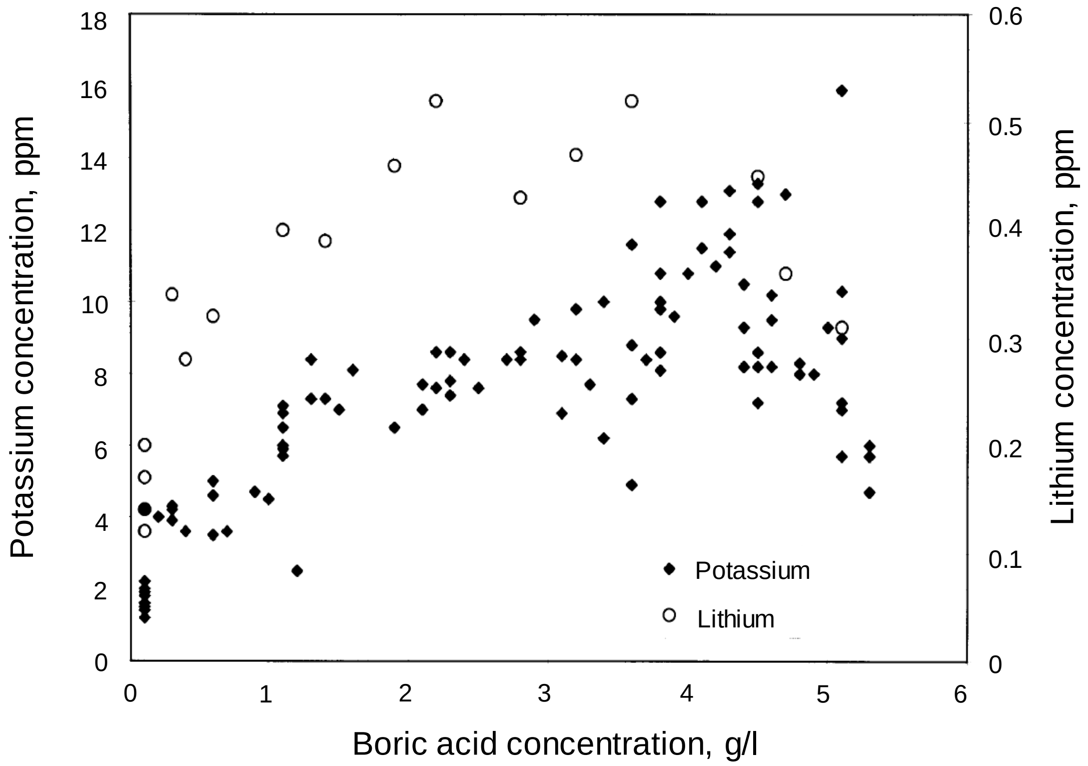
Summary of Chemistry and Radiation Safety Work

- Evaluate primary chemistry pH control, i.e., CVCS bed operation, with multiple alkali
  - Potassium, Lithium (from ${}^{10}_{5}B + n \rightarrow {}^{7}_{3}Li + \alpha$)
  - Sodium (known contaminant from VVER OE)
  - Ammonia
- NSSS vendor review of potential primary system impacts
  - More detailed reviews/assessments if needed
- Assess KOH purity (e.g., sodium) and impact on radio-isotopic generation
- Improve MULTEQ Database with respect to potassium chemistry
  - Model bulk (evaluate control bands) and crevice chemistry (inputs for fuel and materials testing)
- Evaluate VVER operating experience pertaining to:
  - Radiation fields
  - Activated species coolant behavior
  - Effluents and radioactive waste
  - Dose to the public



Adds Complexity to pH_T Control: Now Li and K. New activation species to manage.

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Detailed Plan for Qualifying KOH Updated (Summary)

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High Level Summary of Final Plan (Endorsed by KOH Advisory Committee)

Initiation Testing

Material	Nominal Chemistry (BOC, EOC)	Crevice Chemistry
Nickel-base Alloys	YES	YES
Non-Irradiated SS	NO (when well-controlled) Sufficient VVER OE Yes (in off-normal oxygenated condition)	YES (No oxygen in crevice water chemistry)
Irradiated SS	YES (one chemistry pair: 2 ppm Li and equivalent KOH)	NO
Low alloy steel	NO Sufficient VVER OE	NO Sufficient VVER OE

Crack Growth Rate Testing

Material	Nominal Chemistry (BOC, EOC)	Crevice Chemistry
Nickel-base Alloys	YES	NO Not used to disposition
Non-Irradiated SS	NO Not used to disposition	NO Not used to disposition
Irradiated SS	YES May be used in the future for disposition	NO Not used to disposition
Low alloy steel	NO Sufficient VVER OE	NO Sufficient VVER OE

Material Qualification Testing Summary

- Final Materials testing plan has been completed
  - KOH Advisory Committee endorsed final plan on Sep 26, 2017 webcast
- Crack initiation testing of Ni-base alloys (nominal chemistry)
  - Contract submitted for execution, testing will begin in 2017
- All other testing needs to begin in 2018
  - Irradiated Testing
    - Stainless Steel Initiation and CGR (nominal chemistry)
  - Non-irradiated Testing
    - Stainless Steel Initiation (nominal and crevice chemistry)
    - Nickel-base initiation (crevice chemistry) and CGR (nominal chemistry)

Detailed Plan for Qualifying KOH (Overview)

Qualification Actions Required Prior to Start of Demonstration

Area	Topic	Actions	Schedule
PSCR	Materials Testing	• Initiation & Crack Growth Rate Testing – Non-irradiated testing • Stainless Steel, Alloy 600 – Irradiated testing • Stainless Steel	• 2017-2020
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* Plant Trial to follow Qualification (3 cycles of operation with KOH)

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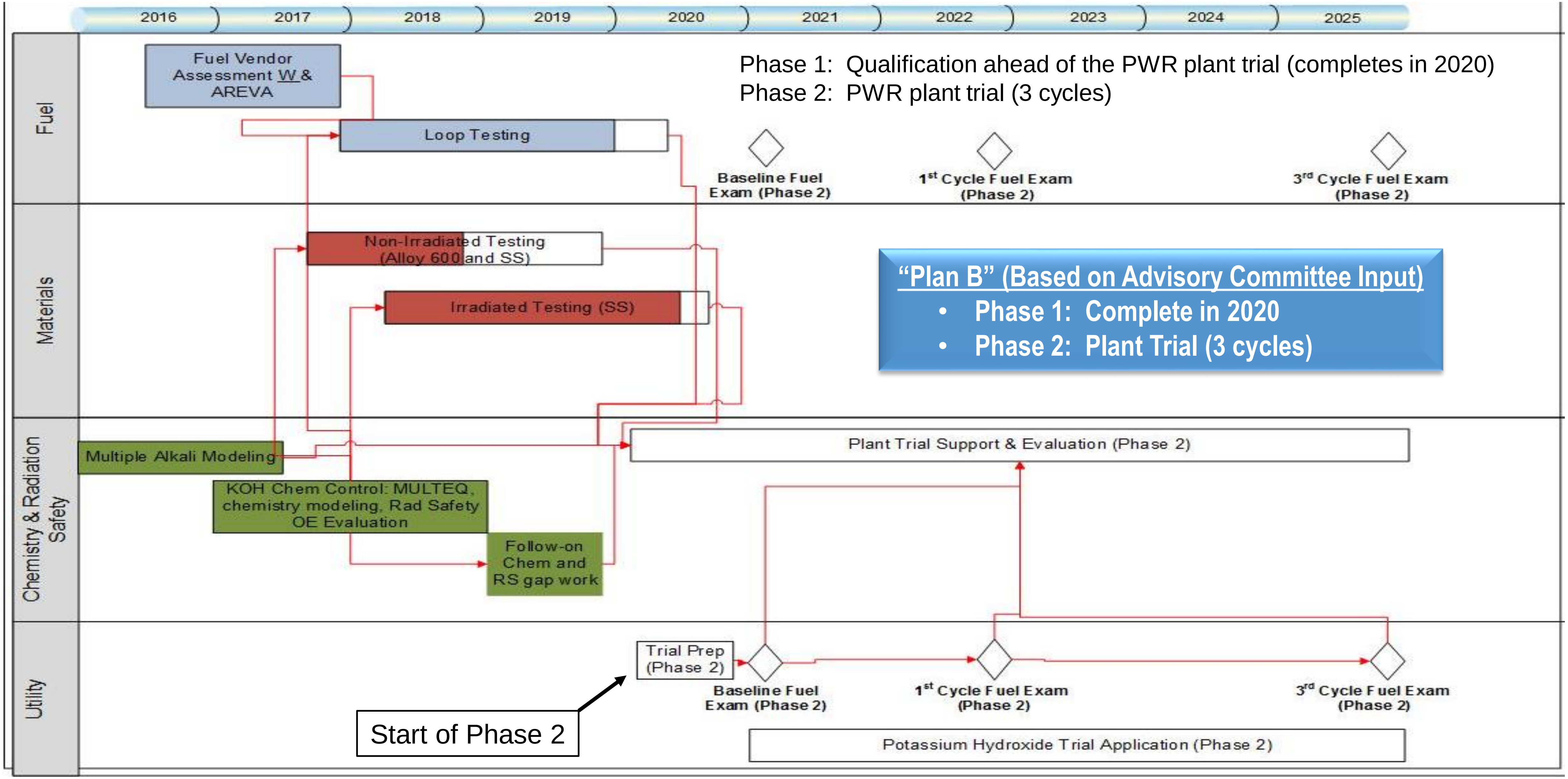
Full Qualification / Demonstration Plan (updated Aug 2017)

Phase 1: Qualification ahead of the PWR plant trial (completes in 2020)
Phase 2: PWR plant trial (3 cycles)



"Plan B" (Based on Advisory Committee Input)
- **Phase 1: Complete in 2020**
- **Phase 2: Plant Trial (3 cycles)**

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Questions?

KOH Qualification

Non-irradiated Materials Testing

Peter Chou
Senior Technical Leader

EPRI-NRC Research Discussion on KOH Qualification
Webcast
October 20, 2017

Project Objective and Approach (Non-Irradiated Materials)

- **<u>Objective:</u>**
  - To conduct due-diligence testing to directly ascertain whether or not a KOH plant demonstration will cause more materials-related damage to the plant related to SCC initiation than its current LiOH chemistry
- **<u>Approach:</u>**
  - Crack initiation testing
  - Crack growth rate testing
  - Compare behavior between LiOH-based and KOH-based water chemistries
    - Startup [at the same pH(T)]
    - Shutdown [at the same pH(T)]
    - Crevice [at different pH(T) because of different concentration factor]
  - KOH is acceptable if it is no worse than LiOH
    - “Better” would be a bonus

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Irradiated SS	YES (one chemistry pair: 2 ppm Li and equivalent KOH)	NO
Low alloy steel	NO Sufficient VVER OE	NO Sufficient VVER OE

Crack Growth Rate Testing

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Irradiated SS	YES May be used in the future for disposition	NO Not used to disposition
Low alloy steel	NO Sufficient VVER OE	NO Sufficient VVER OE

(Non-irradiated) Nickel Alloys: Crack Initiation

- VVERs do not use Ni-base alloys as structural materials
  – Alloy 600 will be used to represent Ni-base alloys
- Crack *initiation* tests of Alloy 600 in nominal PWR primary chemistry are planned
  – Relevant, for example, to steam generators
- Crack *initiation* tests of Alloy 600 in crevice chemistries are planned
  – Crevices are associated with heat-transfer surfaces (specifically, pressurizer heaters)
    - Not all CE pressurizers that use Alloy 600 heater sleeves have been replaced
  – Alkali (LiOH or KOH) can increase due to boiling concentration, resulting in elevated pH that can adversely affect materials
    - Potassium compounds more soluble $\rightarrow$ pH of crevice may be driven higher when KOH is used (vs. LiOH)
    - Difference in pH of crevice likely more influential than K^+ vs. Li^+

(Non-irradiated) Nickel Alloys: Crack Propagation

- Crack *propagation* tests of Alloy 600 in nominal PWR primary chemistry are planned
  - Relevant to thick-section components and their penetrations and nozzles
    - RCS components and piping
    - Reactor vessel heads
- Crack *propagation* tests Alloy 600 in simulated crevice chemistry are not planned
  - Industry does not use crack propagation rates associated with crevice chemistry to disposition OE

High Level Summary of Final Plan (Endorsed by KOH Advisory Committee)

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Crack Growth Rate Testing

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Low alloy steel	NO Sufficient VVER OE	NO Sufficient VVER OE

(Non-irradiated) Stainless Steel: Crack Initiation

- No crack *initiation* testing is planned in nominal PWR primary chemistry (low-ECP)
  - Good performance of stainless steels in both PWR and VVER service
- Crack *initiation* tests in off-normal (oxygen-containing) PWR primary chemistry are planned
  - Limited number of problems attributed to *“the development in stagnant areas of aggressive environments with chlorides, concentrated boric acid and trapped oxygen.”* *
    - e.g. TGSCC in canopy seal welds
- Crack *initiation* tests in crevice chemistries are planned (for the same reason as for Ni-base alloys)
  - Crevices are associated with heat-transfer surfaces (specifically, pressurizer heaters)
  - *“There have been 10 failures of 316L stainless steel pressurizer heaters and one failure of a 316 pressurizer heater sleeve… under… end of cycle conditions, boiling at pressurizer heaters can concentrate lithium to high levels without substantial buffering by boric acid, leading to high pH and increased risk of SCC…” **

** Materials Handbook*

(Non-irradiated) Stainless Steel: Crack Propagation

- No crack *propagation* testing is planned in nominal PWR primary chemistry (low-ECP) or in simulated crevice chemistry
  - Feedback from PWROG: *“We do not currently need CGR testing of non-irradiated stainless steels to support our PWROG work or utility disposition of stainless steel OE at this time.”*

High Level Summary of Final Plan (Endorsed by KOH Advisory Committee)

Initiation Testing

Material	Nominal Chemistry (BOC, EOC)	Crevice Chemistry
Nickel-base Alloys	YES	YES
Non-Irradiated SS	NO (when well-controlled) Sufficient VVER OE Yes (in off-normal oxygenated condition)	YES (No oxygen in crevice water chemistry)
Irradiated SS	YES (one chemistry pair: 2 ppm Li and equivalent KOH)	NO
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Crack Growth Rate Testing

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Nickel-base Alloys	YES	NO Not used to disposition
Non-Irradiated SS	NO Not used to disposition	NO Not used to disposition
Irradiated SS	YES May be used in the future for disposition	NO Not used to disposition
Low alloy steel	NO Sufficient VVER OE	NO Sufficient VVER OE

RPV Steel - PWRs

- Impact of KOH vs. LiOH on RPV steels not an issue for PWR RPVs internally clad with stainless steel
- A few PWR RPVs have localized unclad regions on their interior surfaces
  - e.g. due to wear or the presence of a half nozzle repair
- Some PWR steam generator primary water channel heads have also been affected by localized cladding failure.
- But bare RPV steel is not expected to be susceptible in nominal low-ECP PWR primary chemistry
- Materials Handbook: "*Tests indicate that SCC (stress corrosion cracking) does not occur of pressure vessel steels under normal PWR reactor coolant conditions, i.e., under conditions with normal PWR reactor coolant chemistry characterized by fully deoxygenated conditions with low ECP (near the hydrogen line on a Pourbaix diagram).*"

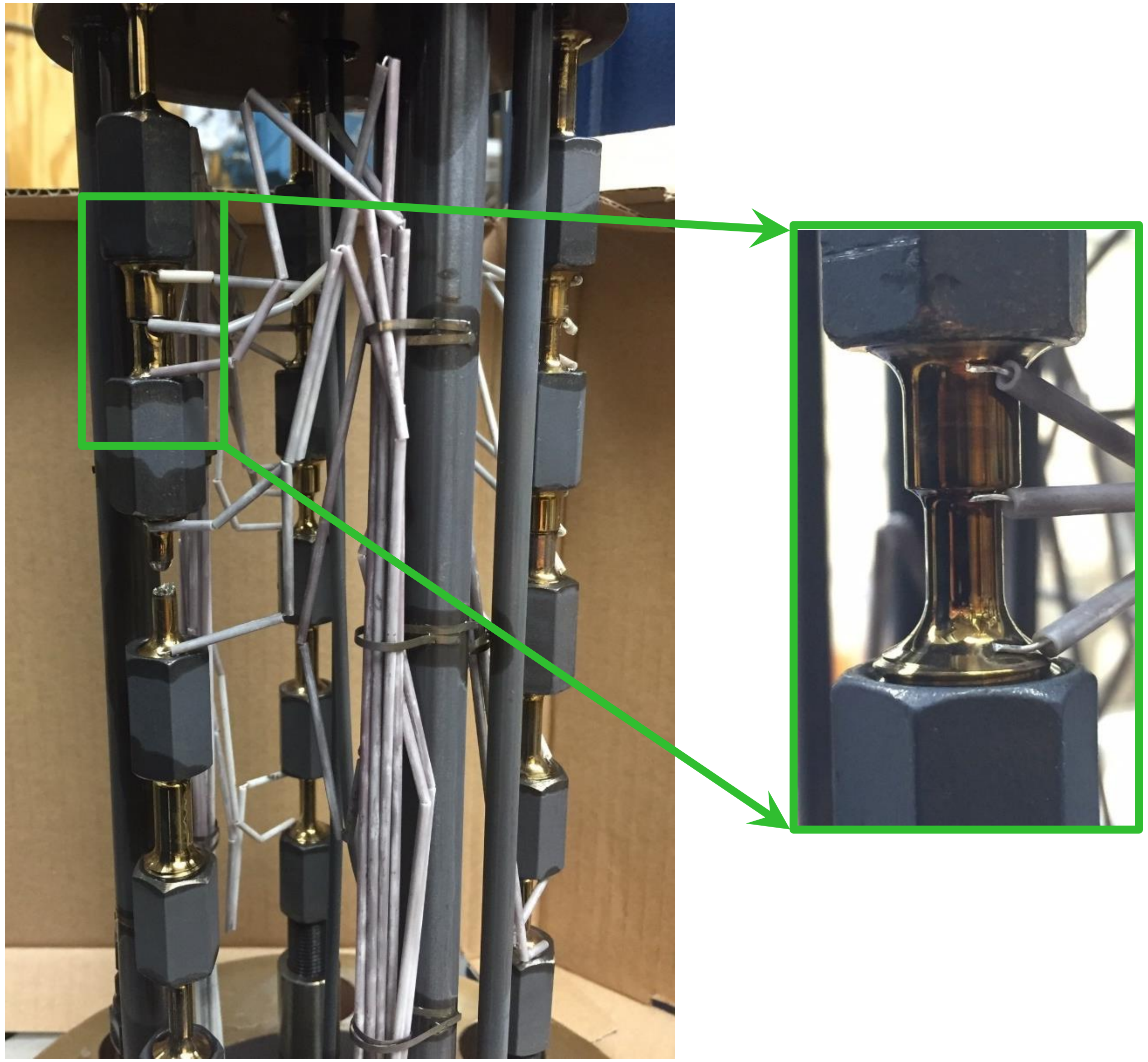
RPV Steel - VVERs

- EPRI's VVER MDM (in review for publication) does not identify SCC, EAF, and environmental reduction of fracture toughness of RPV steel in VVER primary chemistry (based on KOH) as issues for either VVER-440s or VVER-1000s
  - Cites review by Timofeev and Fedorova (1995)
    - Russian RPV steels and their welds have sufficiently high SCC resistance in well-controlled VVER primary water in which a low oxygen concentration is maintained under both design and transient conditions
      - Conclusion considered valid even in the presence of surface damage to overlay stainless cladding in later generation 440 MWe and all 1000 MWe plants
    - VVER-440s inlet and outlet temperatures are 267 ^{o}C and 297 ^{o}C
    - VVER-1000s inlet and outlet temperatures better match PWRs

- Based on the information described, no testing is planned for RPV steels

Crack Initiation: Approach

- Batch Testing
  - Separate autoclaves are required for separate chemistries
  - Larger number of specimens required to obtain statistics of failure for comparison
- Test may be accelerated by higher temperature (e.g. 360°C), cold-work, and/or higher applied stress (e.g. Y.S.)



Crack Initiation: Alloy 600 to Represent Ni-base Alloys

Material: Susceptible Heat of Alloy 600		
Water Chemistry Pair #1	**Water Chemistry Pair #2**	**Water Chemistry Pair #3**
LiOH-based water chemistry #1 (e.g. baseline for startup)	LiOH-based water chemistry #2 (e.g. baseline for shutdown)	LiOH-based crevice chemistry
KOH-based water chemistry #1 of equivalent pH(T) and molarity	KOH-based water chemistry #2 of equivalent pH(T) and molarity	KOH-based crevice chemistry Likely different concentration factor and therefore pH(T)

- Crack initiation testing of Ni-base alloys will be first
- EPRI Water Chemistry Program will provide guidance on needed chemistries
- Susceptible heats of Alloy 600 identified by on-going work at GE GRC (PSCR) and PNNL (MRP)
  - e.g. Heat NX6106XK-11 (MA+15%CF)
- ~10 specimens per autoclave

Crack Initiation: Stainless Steel

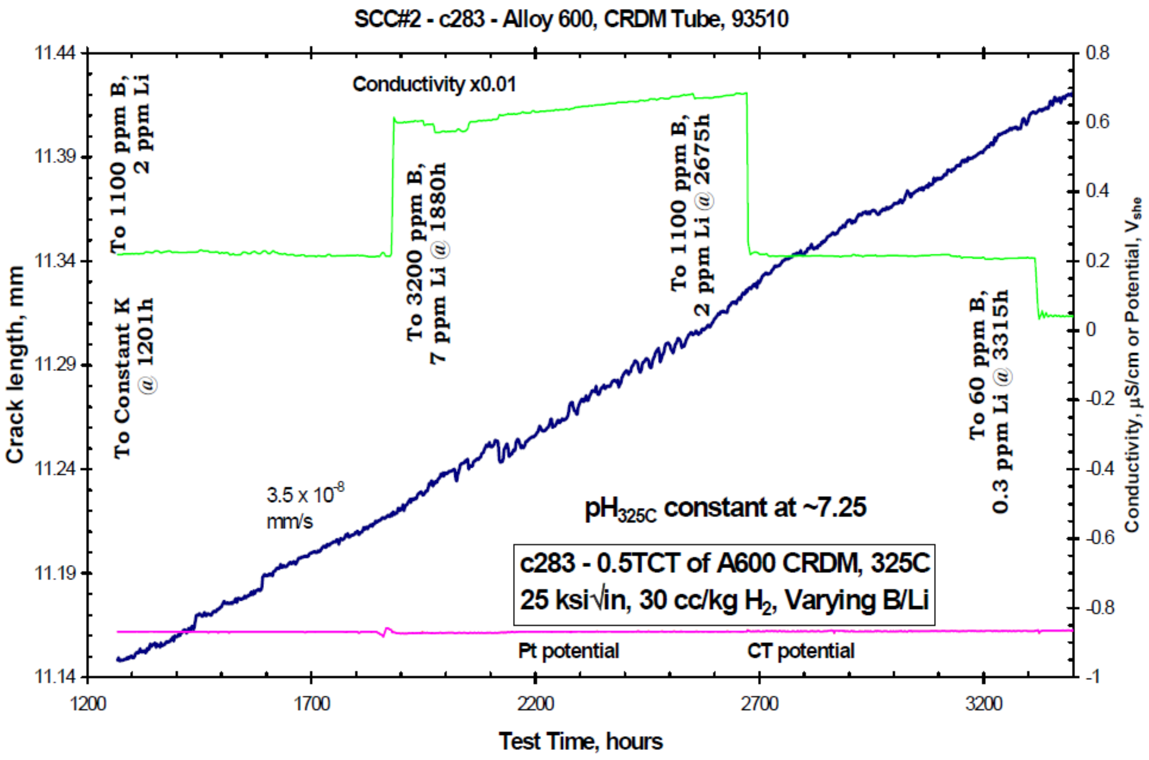
Material: Sensitized and/or 20% CW Stainless Steel	
Water Chemistry Pair #1	
LiOH-based water chemistry #1 containing oxygen to simulated occluded environments	LiOH-based crevice chemistry (No oxygen)
KOH-based water chemistry #1 containing oxygen to simulated occluded environments	KOH-based crevice chemistry Likely different concentration factor and therefore pH(T) (No oxygen)

- Non-irradiated SS, even when sensitized or CW, is resistant to crack initiation in the low-ECP PWR environment, except in
  - Occluded, oxygen-contaminated environments (e.g. TGSCC in canopy seal welds)
    - Testing will not include other contaminants such as Cl^- and sulfate because they complicate interpretation of Li^+ vs. K^+
  - Crevice chemistry (e.g. pressurizer heater crevices)

Crack Propagation: Approach

- The changes in water chemistry will be implemented on the fly
  - New water chemistry will be introduced to displace the previous water chemistry without changing any other test parameter
    - Monitor (differences in) crack growth rates
    - Methodology applied in: *Materials Reliability Program: Effects of B/Li/pH on PWSCC Growth Rates in Ni-Base Alloys (MRP-217). EPRI, Palo Alto, CA: 2007. 1015008.*
- Two CGR specimens will be tested in tandem in one test train
  - Assumption: The pairs of specimens have sufficiently similar CGRs to be tested in tandem
- Test may be accelerated by higher temperature (e.g. 360ºC), cold-work, and/or higher stress-intensity factor

Example of On-the-Fly Changes in Water Chemistry



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Crack Propagation: Alloy 600

- Water Chemistry Sequence:

  Reference LiOH #1 → KOH-based water chemistry #1 →
  Reference LiOH #2 → KOH-based water chemistry #2 →
  Reference LiOH #3 → KOH-based water chemistry #3

- The different water chemistry pairs can represent startup, operation, shutdown
  - The pairs will be at equivalent pH(T) and molarity

Questions?

KOH Qualification

Irradiated Stainless Steel Testing

Jean Smith, PhD, PE
Principal Technical Leader

EPRI-NRC Research Discussion on KOH Qualification
Webcast
October 20, 2017

IASCC Mechanisms

Austenitic stainless steel PWR reactor internals components are susceptible to irradiation-assisted stress corrosion cracking (IASCC)

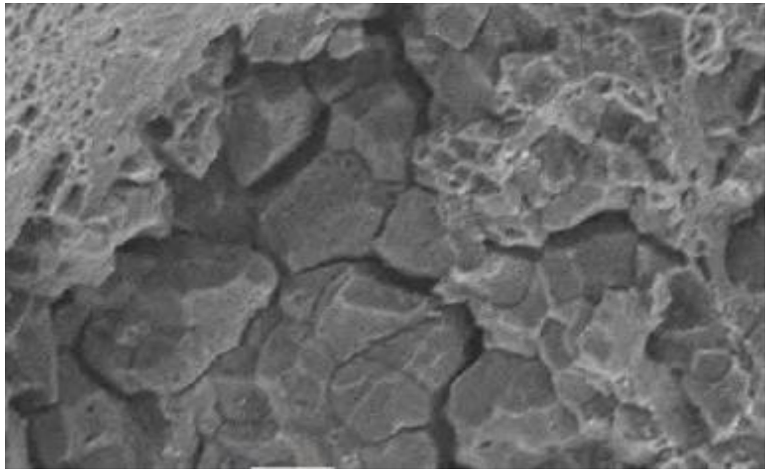
- Baffle and former plates
- Core barrels and welds
- Bolts

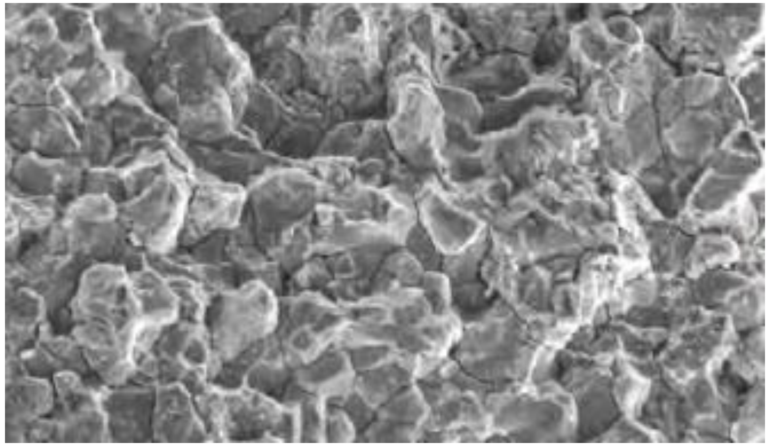
All stress corrosion cracking mechanisms require three conditions to be met simultaneously

- A susceptible **material** must be exposed to a corrosive **environment** and subjected to a tensile **stress**

Mechanisms proposed to explain IASCC are typically categorized as environmental or material

- **Environmental**: Radiation water chemistry → changes in corrosion potential or solution conductivity
- **Material**: Neutron irradiation → microstructural (radiation hardening and creep) and microcompositional (radiation-induced segregation of impurities and redistribution of major alloying elements) effects

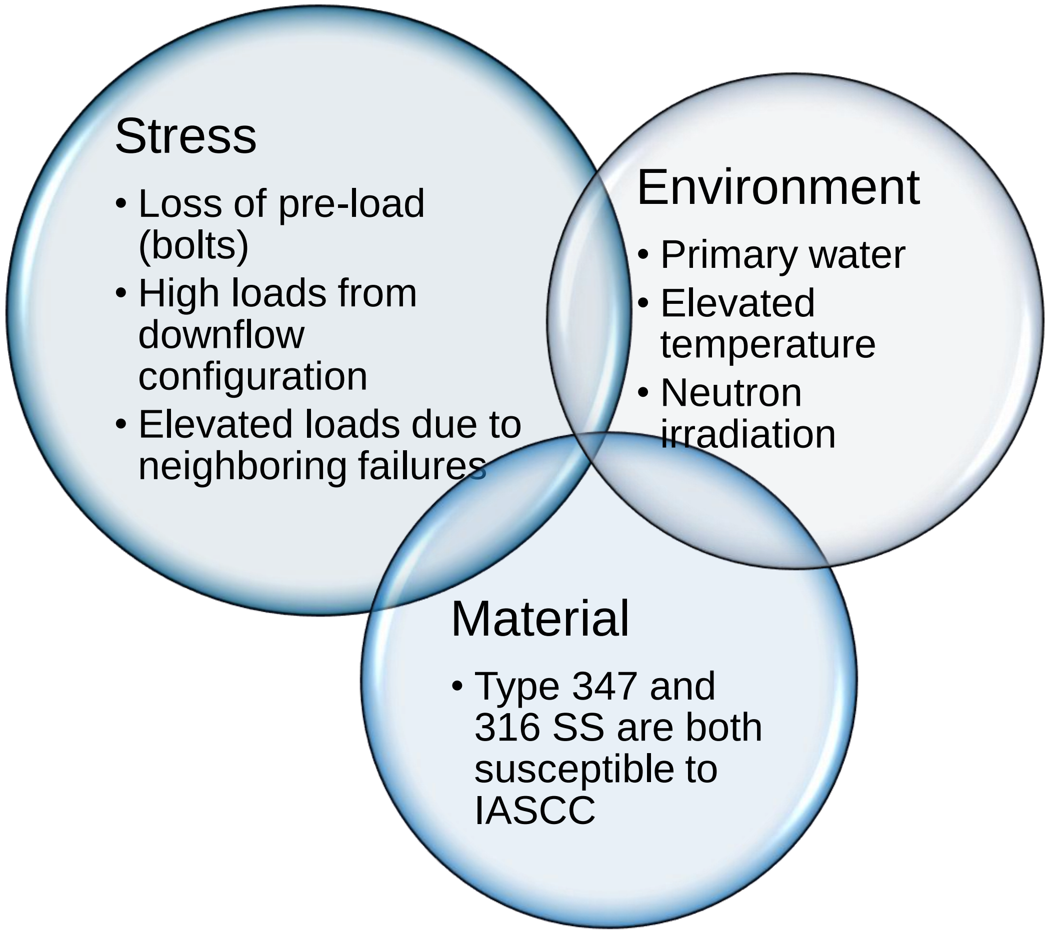




IASCC Operating Experience

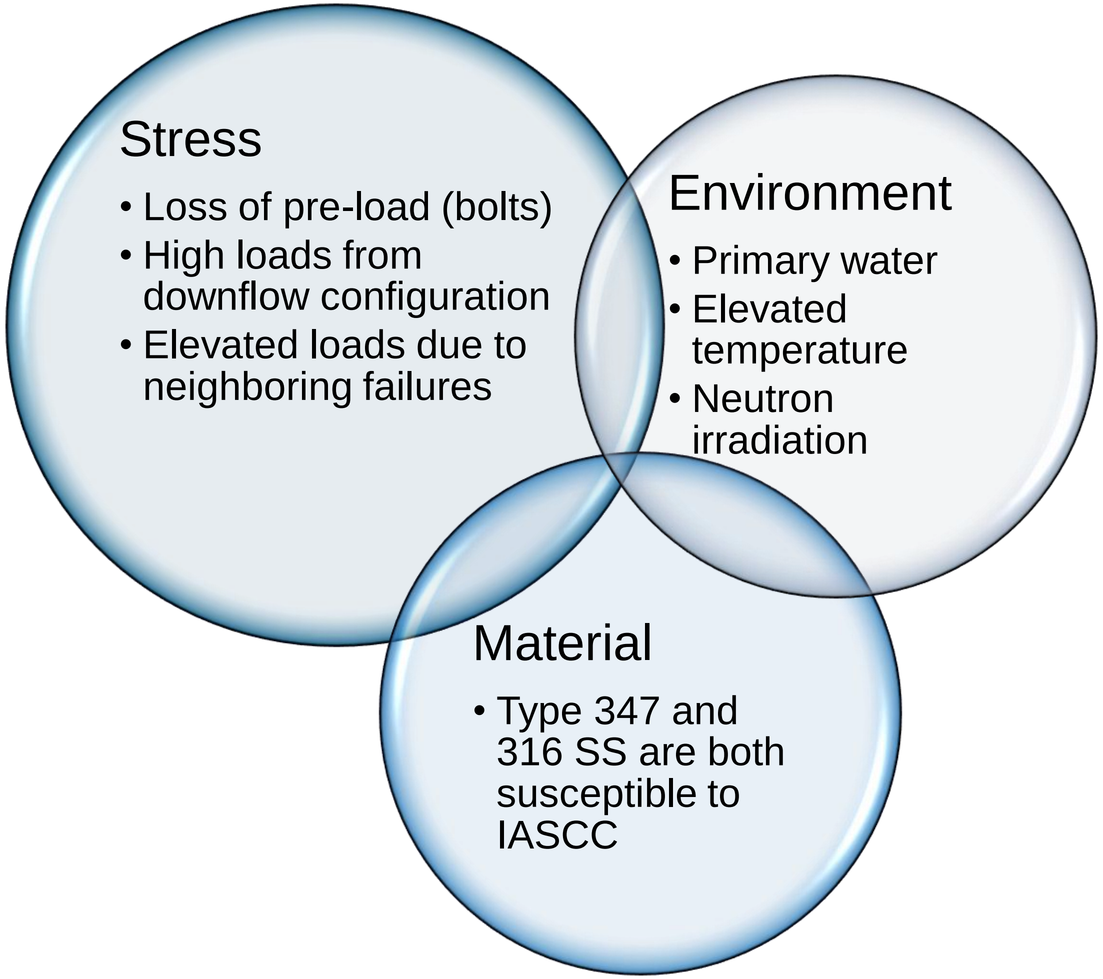
Austenitic stainless steel baffle-to-former bolts (BFBs) represent the largest population of reactor internals components with degradation by IASCC

- **Material**
  - VVERs use titanium-stabilized austenitic stainless steels, which are more resistant to SCC than non-stabilized stainless steels
    - PWRs: BFBs made from both Types 316 and 347 SS have experienced failures from IASCC
    - VVERs: Titanium-stabilized austenitic SS BFB failures have occurred
  - The threshold stress dependence curve for IASCC initiation of VVER reactor internals materials as a function of neutron fluence is based on PWR data.



IASCC Operating Experience

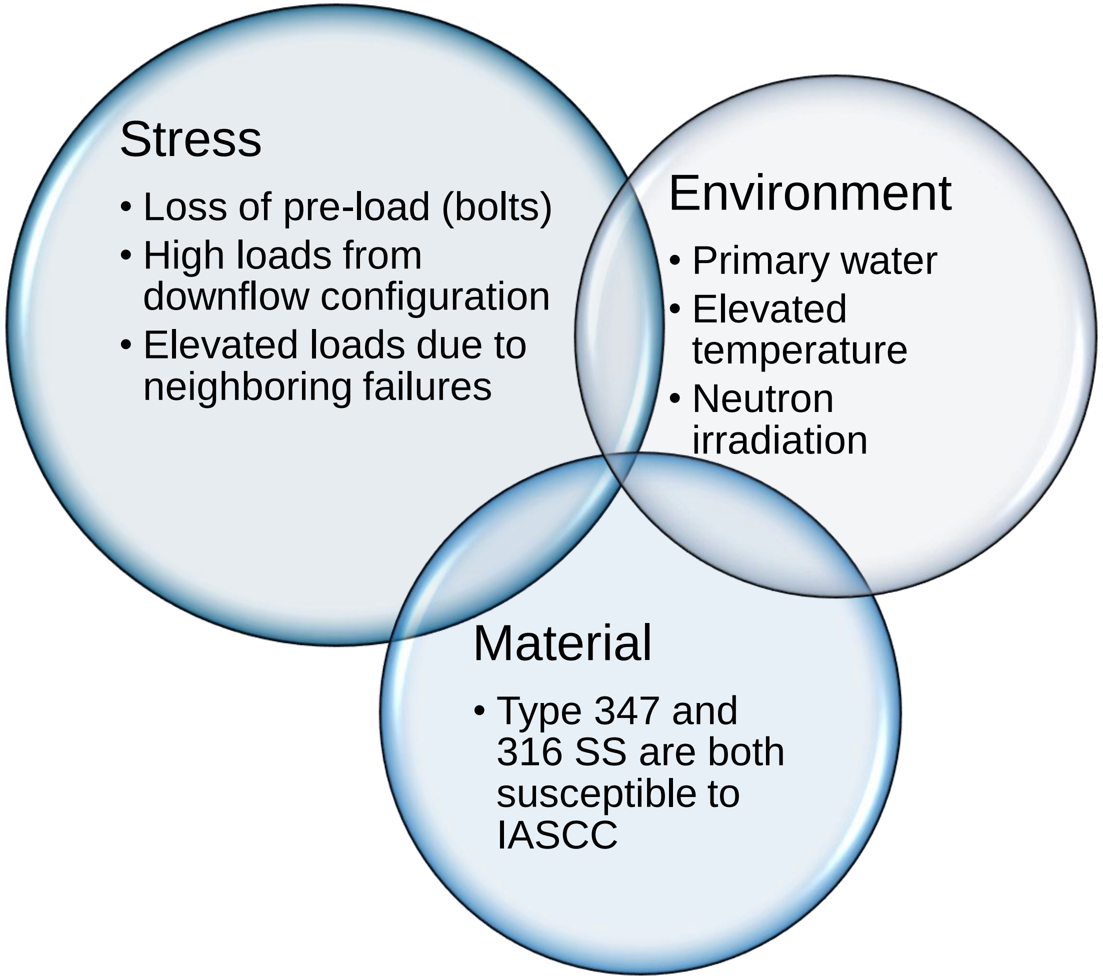
- **Environment**
  - High concentrations of KOH or LiOH are not likely on reactor internal surfaces (other than fuel) due to the lack of heat flux, which is required for concentration to occur
    - Potential for a crevice chemistry to develop as the result of localized areas of gamma superheating in the region beneath the bolt head
  - IASCC failures in BFBs examined in the lab have initiated in the high-stress head-to-shank region
  - In isolated (non-cluster) bolt failures, cracking has initiated and propagated by typical IASCC mechanisms consistent with the load, dose, and effective full power years (EFPY) of the components
    - Stochastic nature of BFB IASCC failures suggests localized chemistry changes are not primary driver



IASCC testing will be conducted in bulk chemistry environments

IASCC Operating Experience

- **Stress**
  - Changes in the stress state of BFBs due to loss of pre-load and/or failure of neighboring bolts
  - IASCC has been observed as the initiating cause of BFB failures, and in “single-event” failures, it is also the propagating mechanism
  - In clustered BFB failures, stress plays a leading role in both crack initiation and propagation
    - Crack propagation morphologies observed on failed bolts include:
      - IASCC
      - Transgranular cracking
      - Fatigue
      - Ductile overload



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Irradiated Stainless Steel – Crack Initiation Testing

Existing data on crack initiation of irradiated stainless steel in LiOH can be used for benchmarking KOH effects

- MRP Lithium Effects Testing
  - Effect of LiOH concentration on IASCC initiation rate as a function of applied stress in high dpa material
  - Used Type 316 CW flux thimble tube material at 60 and 100 dpa (both O-ring and UCL specimens)
  - Li levels of 2.0 and 8.0 ppm at $pH_{300°C}$= 7.2, 340°C, 30 cc/kg DH
  - *At high LiOH level: cracks initiated in a shorter amount of time and at lower stress levels*
- MRP Zorita Internals Research Project
  - Zorita baffle plate material (Type 304 SS) at ~45 dpa
  - Environment: 2 ppm Li, 1000 ppm B, 30 cc/kg DH, 320°C
  - *Failure of UCL specimens more prevalent*

Irradiated Stainless Steel – Crack Initiation Testing

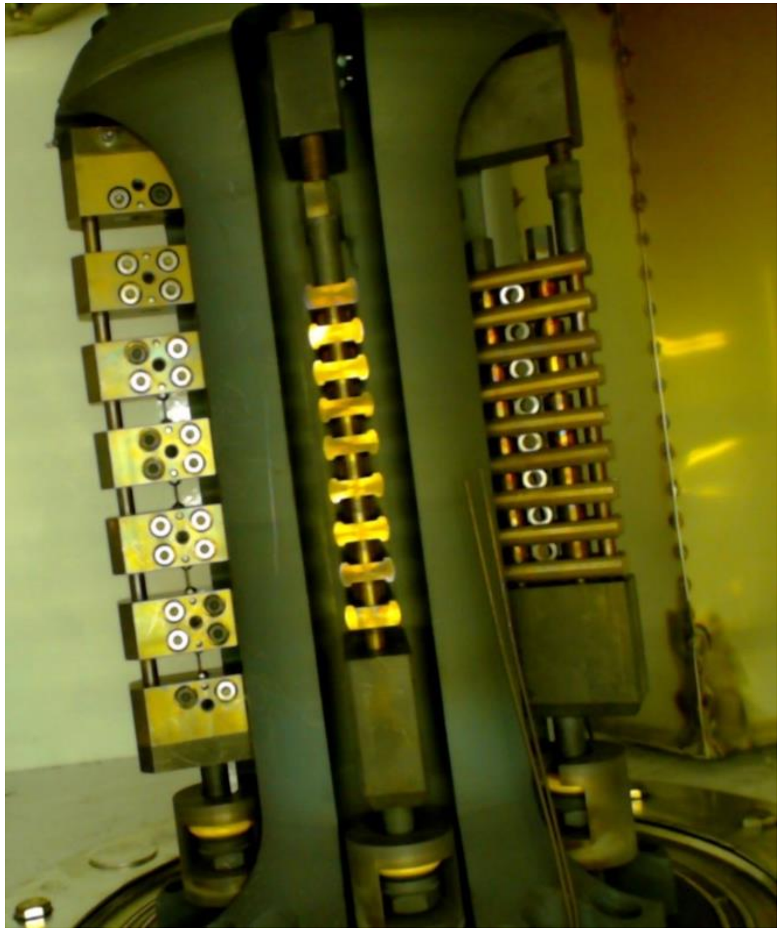
Objective: Determine the relative effect of KOH-based water chemistries on IASCC crack initiation compared to the equivalent LiOH environments

Test Material
- Type 316CW flux thimble tube ~60 dpa **–OR–**
- Type 304 baffle plate ~40 dpa

Specimen Type
- O-ring specimens (Type 316)
- Uniaxial constant load (UCL) specimens (Type 304)

Test Duration
- 4,500 to 6,000 hours (+ specimen prep and post-test exams)
- Consistent with MRP-211 database

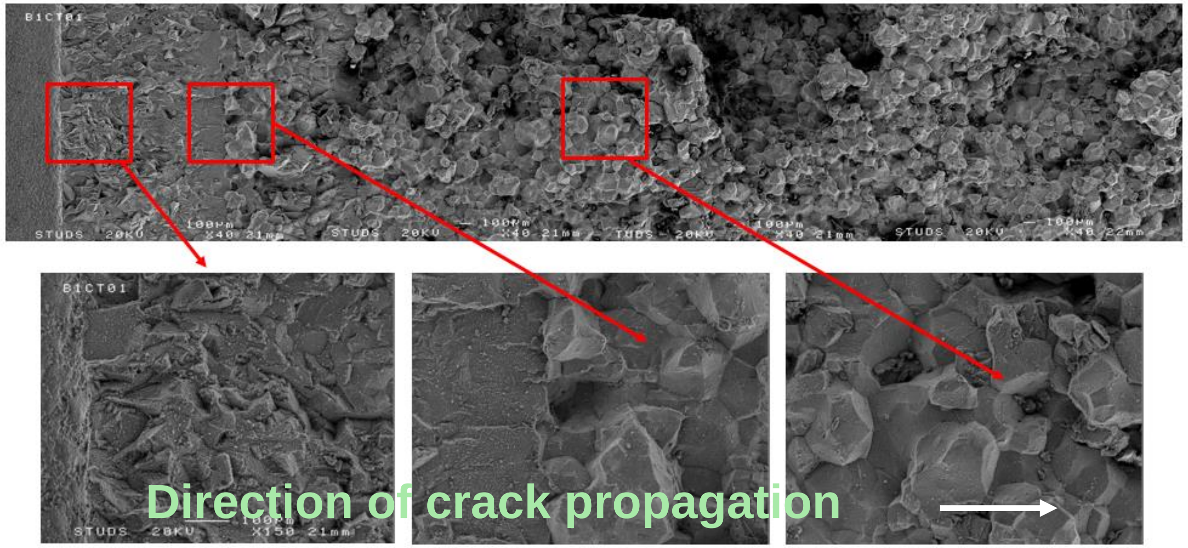
Test Environment
- 320 to 360°C, KOH-based water chemistry equivalent to 2.0 to 3.5 ppm Li (pH_T and molarity)



Irradiated Stainless Steel – Crack Growth Rate Testing

- Data on crack growth rates of highly irradiated stainless steel in LiOH can be used for benchmarking KOH effects
- MRP Zorita Internals Research Project
  - Zorita baffle plate material (Type 304 SS) at 10, 30, and 50 dpa
  - Environment: 2 ppm Li, 1000 ppm B, 30 cc/kg DH, 320°C
  - Crack growth rates at three (3) temperatures and three (3) stress intensity levels for both 30 and 50 dpa
    - *CGRs have been shown to be very low in Zorita baffle plate material*
  - Work to be completed prior to start of KOH qualification program

- Halden Research Program
  - Zorita baffle plate material at ~35 dpa to be tested online at Halden in BWR normal water chemistry, BWR hydrogen water chemistry, and typical PWR water chemistry
  - Work to be completed prior to start of KOH qualification program



Irradiated Stainless Steel – Crack Growth Rate Testing

Objective: Determine the relative effect of KOH-based water chemistries on IASCC crack growth rates compared to the equivalent LiOH environments

Test Material

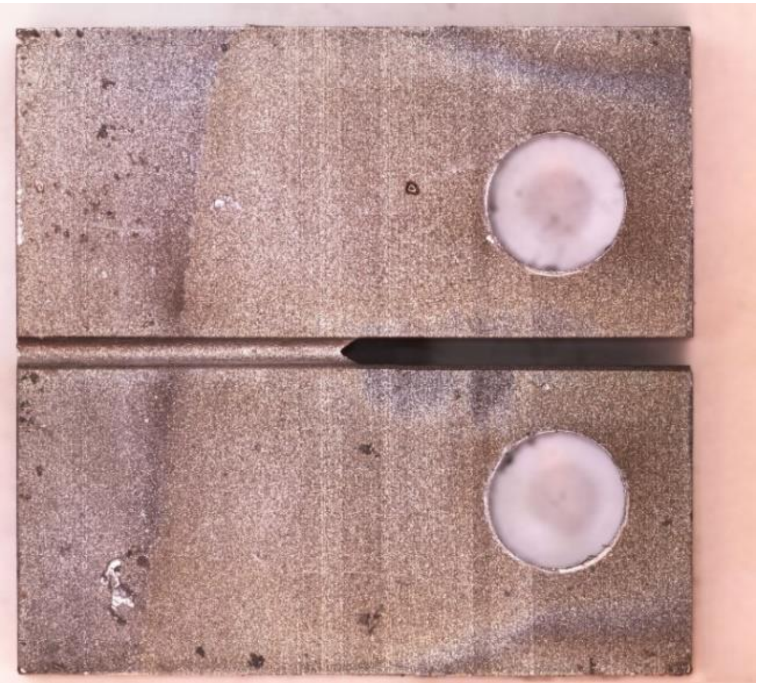
- Zorita Type 304 stainless steel baffle plate material
- Dose range of 30 to 50 dpa allows for comparison with existing program

Specimen Type

- 1/2T-CT specimens
- Specimen is large enough to test multiple environments with one specimen
- One stress-intensity level will be selected based on Zorita results
- One specimen to be tested; *one* specimen for backup

Test Environment

- 320 to 360°C
- *Two (2) KOH-based water chemistries* with analogous LiOH reference chemistry



Questions?

Big Picture

- Chemistry and Radiation Safety (2016–2019)
  - Work is ongoing and fully funded through 2019
- Fuel (2016–2020)
  - Vendor reviews underway, to be completed in 2017
  - Loop testing to start in 2018 and complete in 2020
  - Majority of needed funding has been identified, with remaining funding highly certain
- Materials (2017–2020)
  - Starting crack initiation testing of non-irradiated nickel-base alloy (nominal chemistry)
  - Other testing needs to begin in 2018
    - Irradiated Stainless Steel Testing: ~85% funded
    - Non-irradiated Alloy 600 and Stainless Steel Testing: ~45% funded
    - Strategic Gap Funding recently confirmed to support needed 2018 materials testing work
    - On-going discussions with DOE/LWRS on collaboration
    - Initiated discussion with Materials Ageing Institute (MAI) in France
    - Initiating discussion with NRC Research
- Plan has Qualification effort completed in 2020, with start of Plant Trial in 2021

Together…Shaping the Future of Electricity