SCC Crack Growth Rate Testing of Nickel-Base Alloy 690 and Alloy 152 in PWR Primary Water

Steve Bruemmer and Mychailo Toloczko
Pacific Northwest National Laboratory

NRC Project N6007

Research Supported by
U.S. Nuclear Regulatory Commission
NRC Project Managers:
Hipolito Gonzalez and Eric Reichelt

Battelle

Pacific Northwest National Laboratory

Project Objective & Scope

Conduct SCC crack-growth-rate (CGR) testing of nickel-base stainless alloys focused on Alloy 690 and its weld metals Alloy 152 and 52. Other relevant materials may also be included in the required test matrix including materials removed from operating plants. State-of-the-art test systems will be constructed to allow experimentation under simulated and/or accelerated (e.g., increased temperature, more aggressive environments, increased load range or load interaction effects) PWR and BWR conditions. Direct current electric potential drop methods will be used to acquire crack extension data and reference electrodes will be used to monitor corrosion potential.

Major Task Areas

- **Crack-Growth System Construction/Testing**
  - CGR systems and validation testing completed in 2006.
- **Crack-Growth Rate Testing**
  - Austenitic Stainless Steels
  - Alloy 600 and Alloy 182 Weld Metal
  - Alloy 690 and Alloy 152/52 Weld Metals
- **Characterization and Cracking Mechanisms**
  - OM, SEM, EBSD and TEM examinations of test materials
  - SEM/ATEM characterizations of SCC cracks and crack tips in LWR service materials and laboratory CGR samples
  - SEM/ATEM characterizations of hot cracks in mockup and laboratory alloy 182/82 and alloy 152/52 weldments

PNNL SCC Crack-Growth Testing

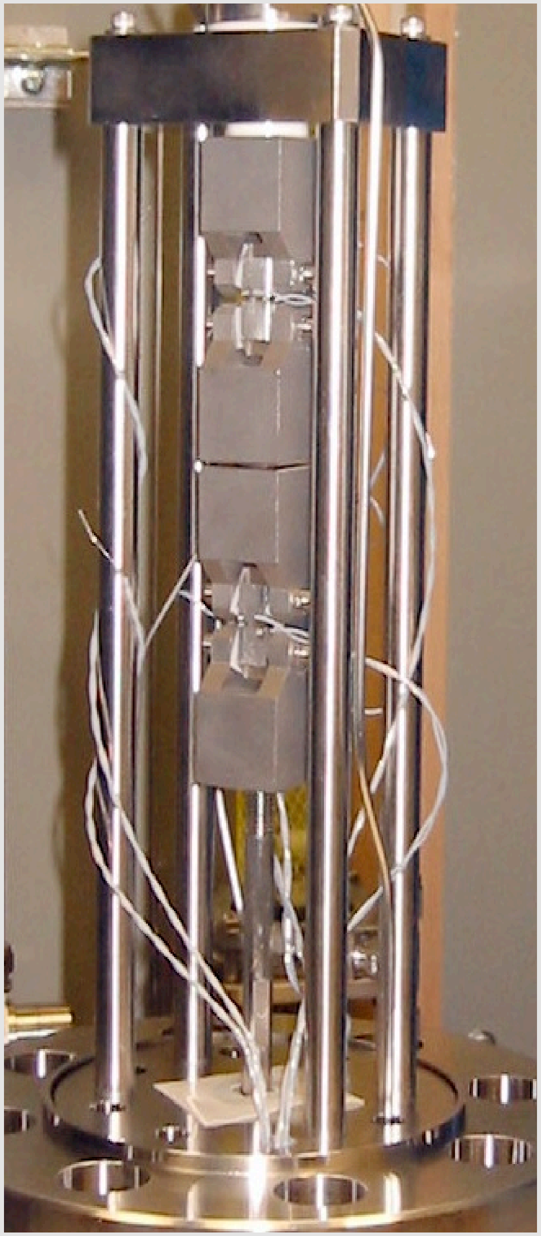
- **Austenitic Stainless Steels**
  - Demonstration/Validation Tests (7) on CW316SSs and sensitized 304SSs in 288°C BWR-NWC and/or BWR-HWC
- **Alloy 600 and Alloy 182**
  - ICG-EAC Round Robin: Alloy 600 and 182 in BWR water; Alloy 182 in 325°C PWR primary water
  - *H_2 and ECP effects on SCC in two alloy 182 welds and on a cold-worked alloy 600 in 325°C PWR primary water*
  - *North Anna 2, nozzle 31, Alloy 182 J-groove weld being machining for SCC testing*
- **Alloy 690 and Alloy 152**
  - *Alloy 152 Weld Metal in 325-350°C PWR primary water; tests on as welded and stress-relieved conditions*
  - *Alloy 690 CRDM heats in 325-350°C PWR water; tests in as-received TT condition; plus material modifications by heat treatment, cold working and 1D cold rolling.*

PNNL SCC Crack Growth Test Systems

- *Outlet conductivity ≤ 0.065 μS/cm under BWR test conditions*
- *Reversing DCPD, automated K control, autoclave flow rate of 220 cc/min.*
- *Continuous measurement of load, inlet conductivity, outlet conductivity, DCPD voltage and current, autoclave water temperature, ECP, and other parameters*
- *Water conductivity in conjunction with manual pH measurement for B/Li monitoring.*

Typical Test Setup & Approach
Alloy 690 and 152

- *PWR Primary Water Conditions: 1000 ppm B, 2 ppm Li, 11-29 cc/kg H_2 and test temperature 325-350°C.*
- *Single sample fatigue precracked in situ, 2-3 samples in series precracked ex situ; typical K = 20-30 MPa√ m*
- *Crack transitioning in situ: decreasing fatigue cycle frequency; to hold time + gentle cycle; finally to constant K.*
- *Crack length measurement calculated from DCPD data using reference DCPD potential correction taken from back face of sample.*



Battelle

PNNL Test Status: October 2007

Alloy 152

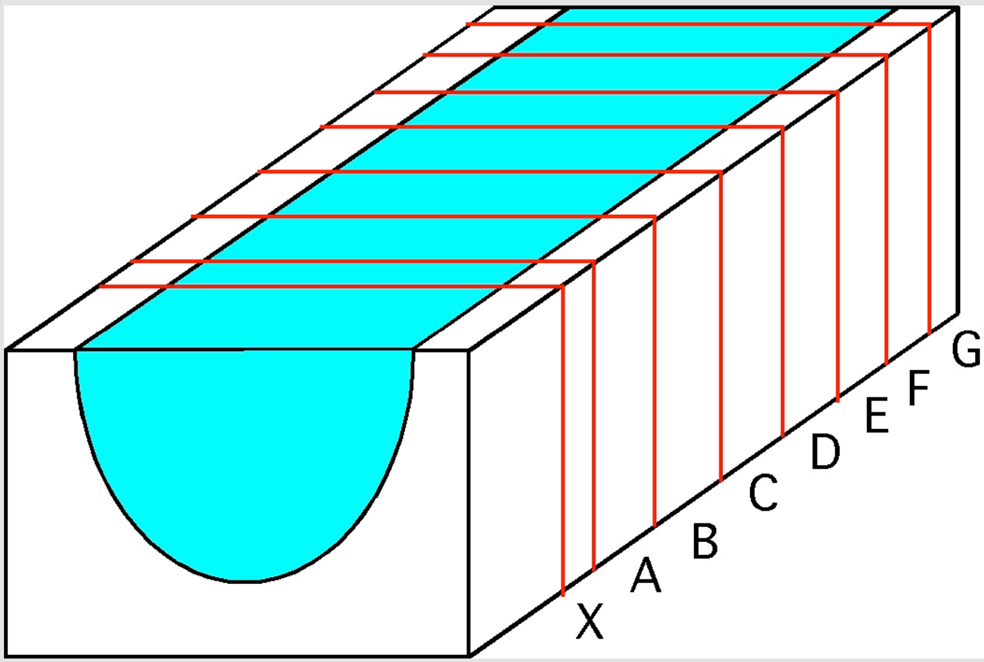
- ***Alloy 152 Weld Metal in 325°C PWR primary water***
- Alloy 152 Weld Metal in 325-350°C PWR primary water; as welded and stress relieved conditions

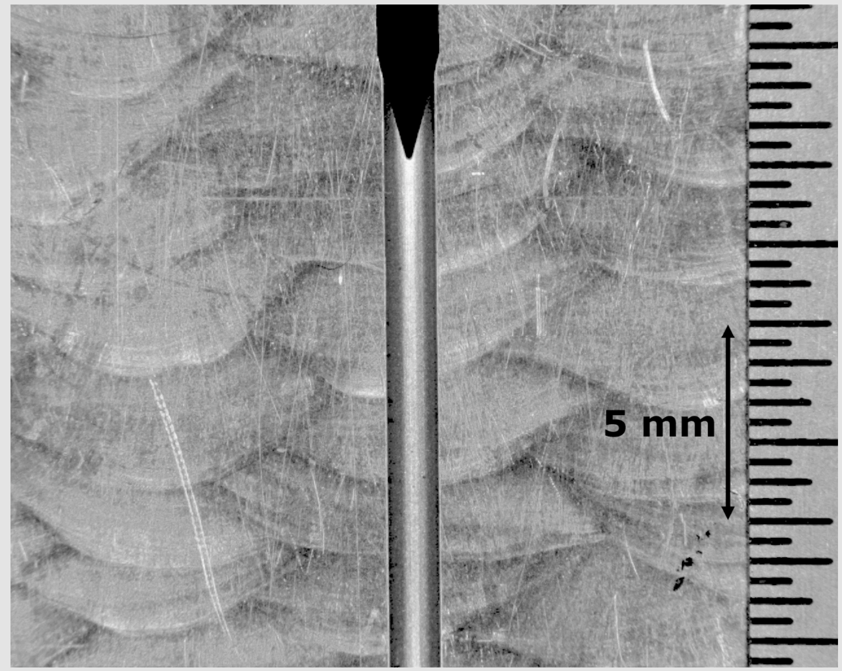
Alloy 690

- Alloy 690 CRDM heat RE243 in 325°C PWR primary water; as-received TT and SA conditions
- Alloy 690 CRDM heat RE243 in 325-350°C PWR primary water; as-received TT and SA conditions; 1D cold rolled to 17% reduction in SL orientation and 1D cold rolled to 17% and 30% reduction in TL orientation
- Alloy 690 CRDM heats RE076, WP140 and WP142 in 350°C PWR primary water; as-received TT condition

Alloy 152 Mock-Up Weld

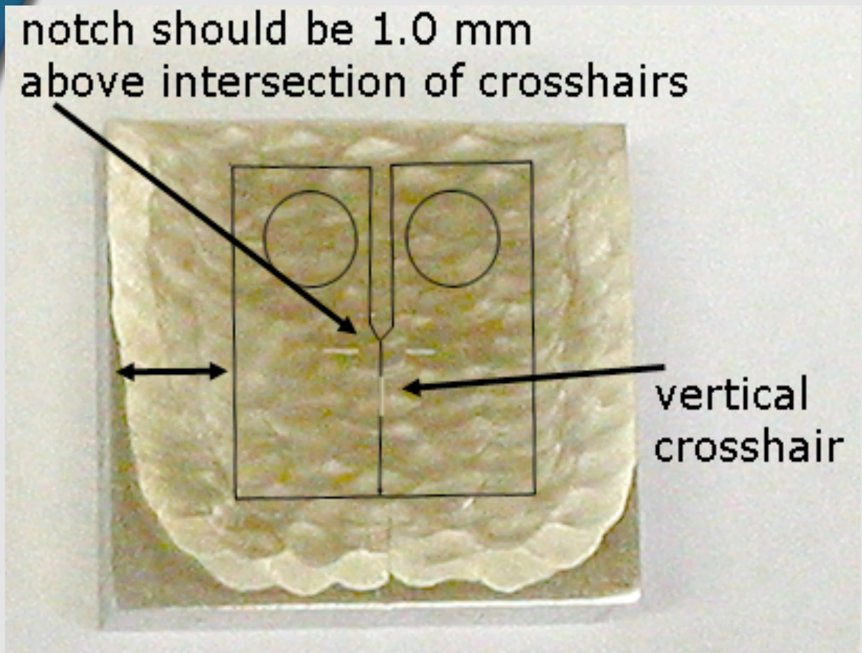
Made using 6.1-cm-thick, 304SS plates, Alloy 152 butter passes and weld obtained from EPRI NDE Center produced by MHI for Kewaunee pressure vessel head replacement

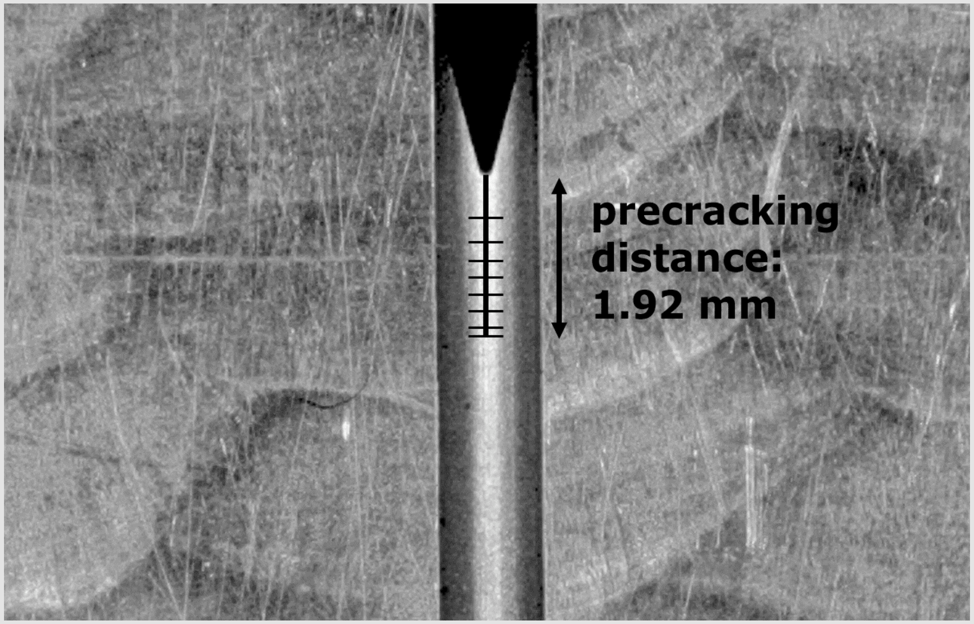




Weldment was sectioned into 1.9-cm thick blocks and surfaces of CT blanks (C, D and E) characterized so that pre-cracks are located in a single weld pass, aligned with elongated dendrites and enable >1 mm of SCC crack growth within the weld pass.

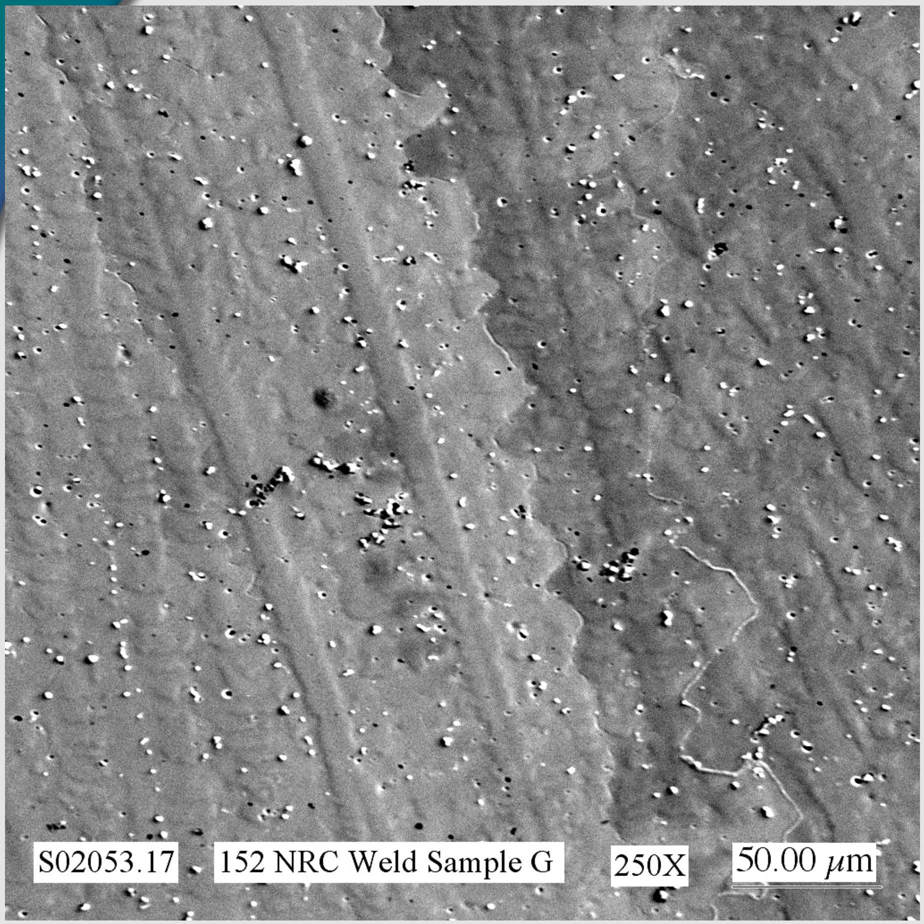
Alloy 152 Weld Metal Specimen

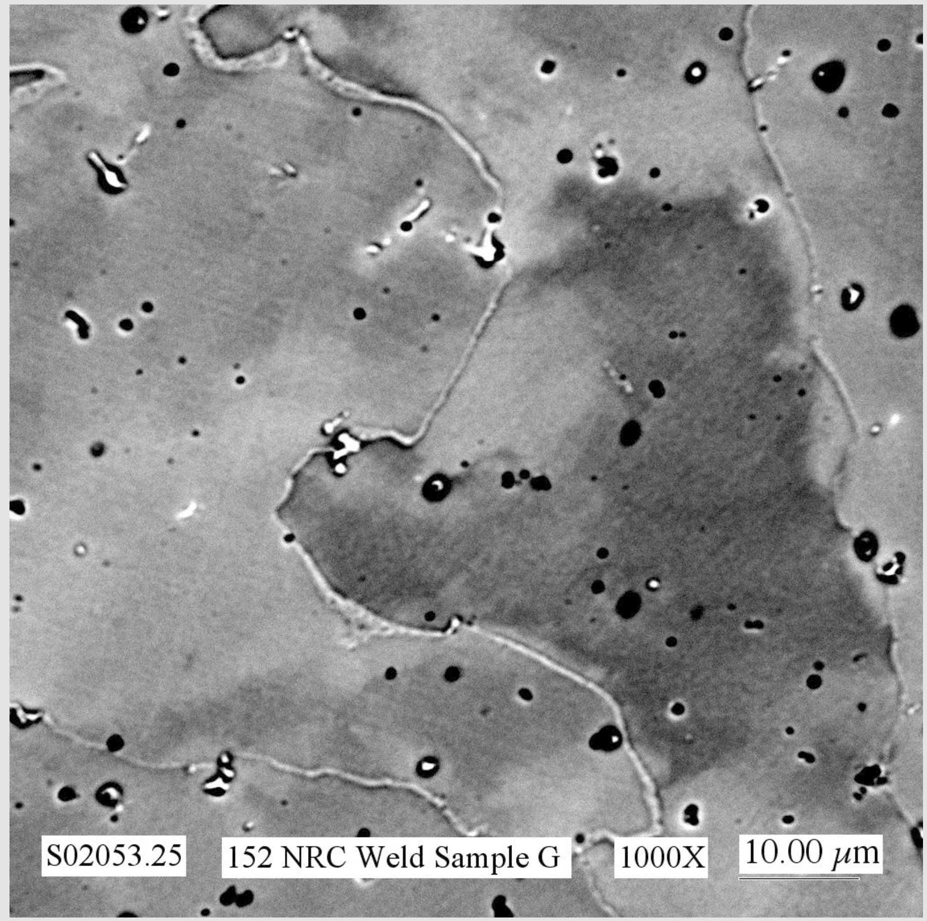




Sample machined entirely of weld metal. Notch and pre-crack oriented to allow SCC growth in the middle of a weld pass with crack oriented roughly along dendrite direction.

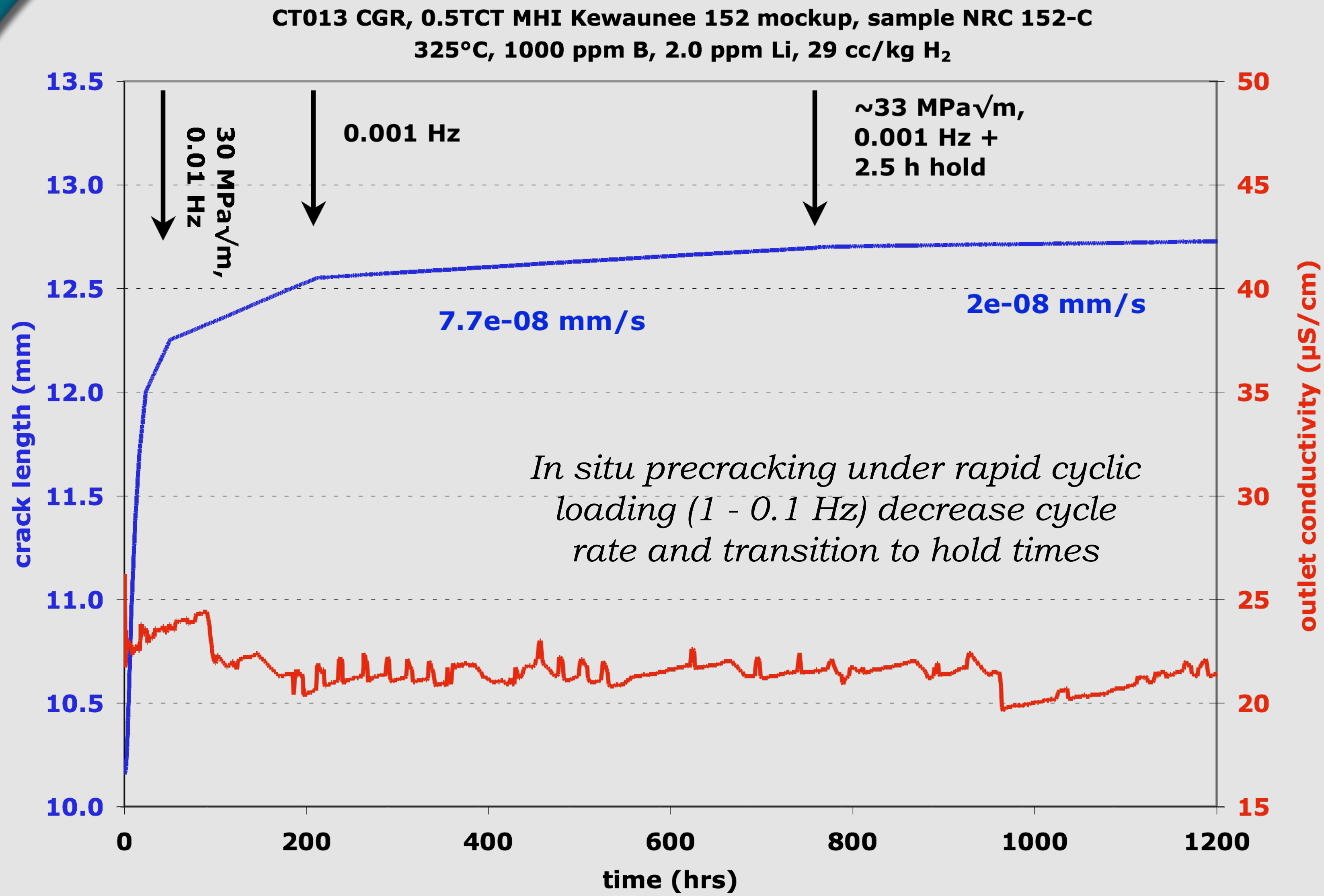
Alloy 152 Weld Metal Microstructure



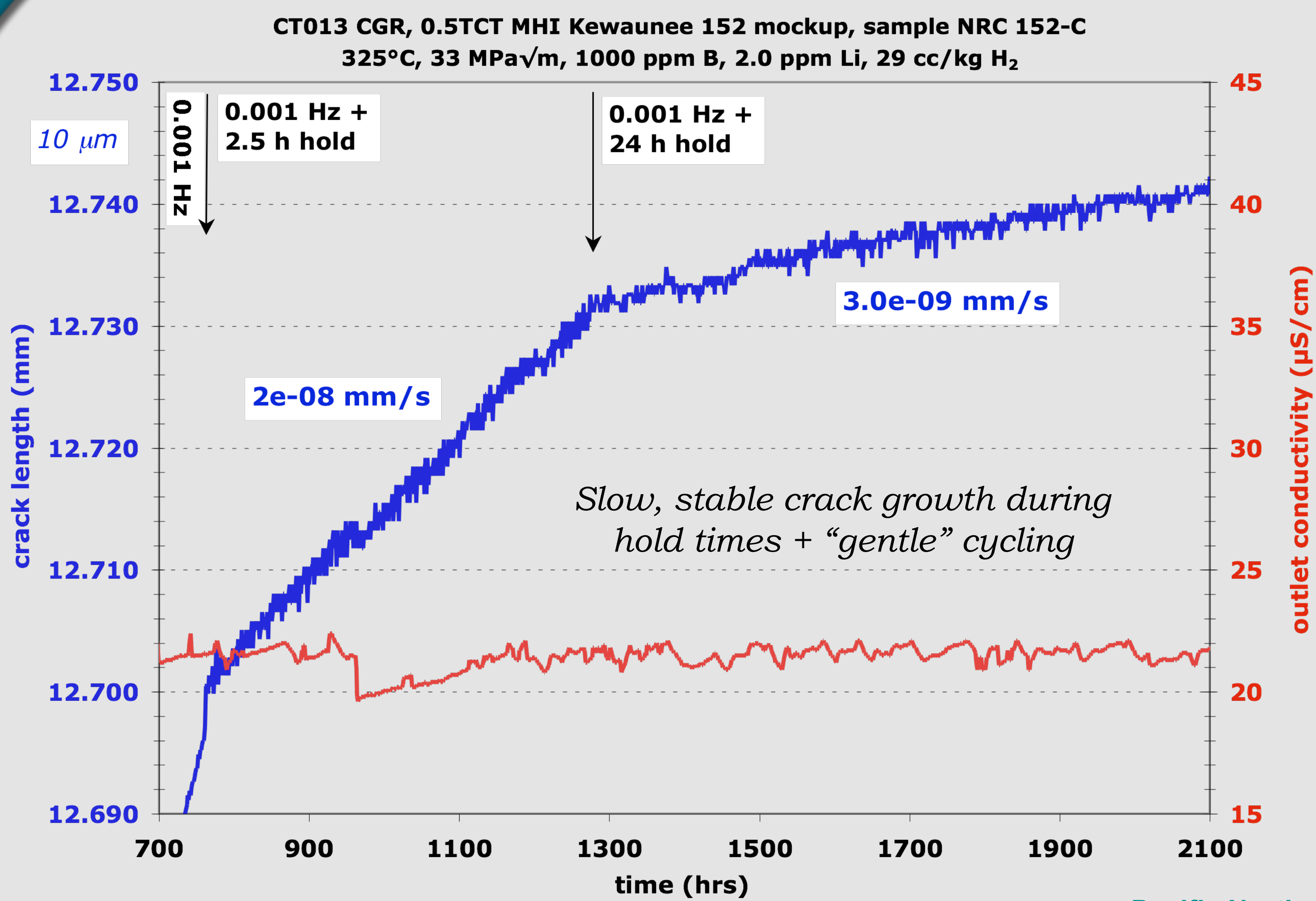


SEM backscatter electron images showing convoluted dendritic boundaries with a high density of Al-Ti-oxide (dark) and Nb-carbide (light) particles throughout the matrix. Fine grain boundary carbides on many grain boundaries.

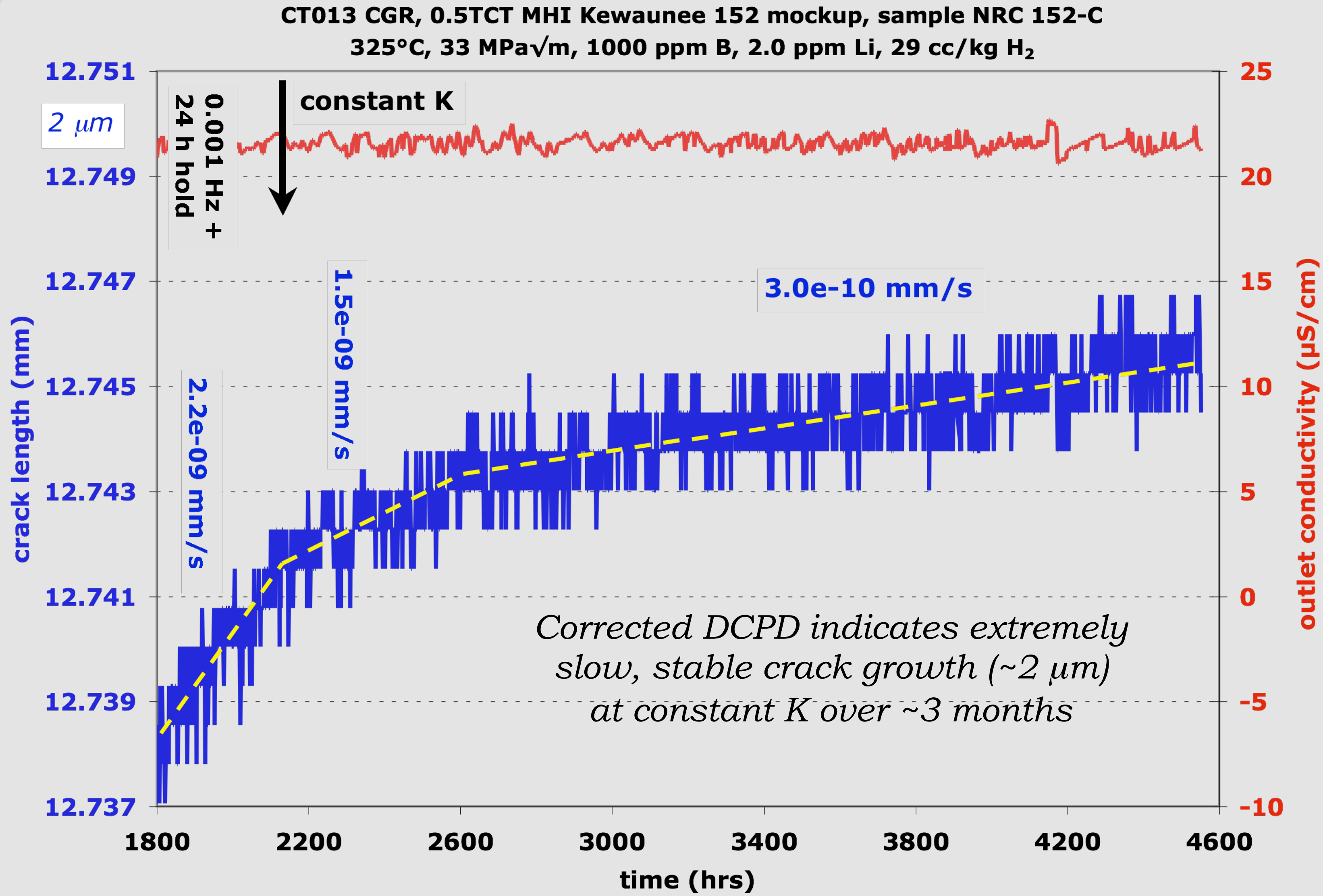
Alloy 152
Precracking and Transitioning



Alloy 152: Cycle + Hold

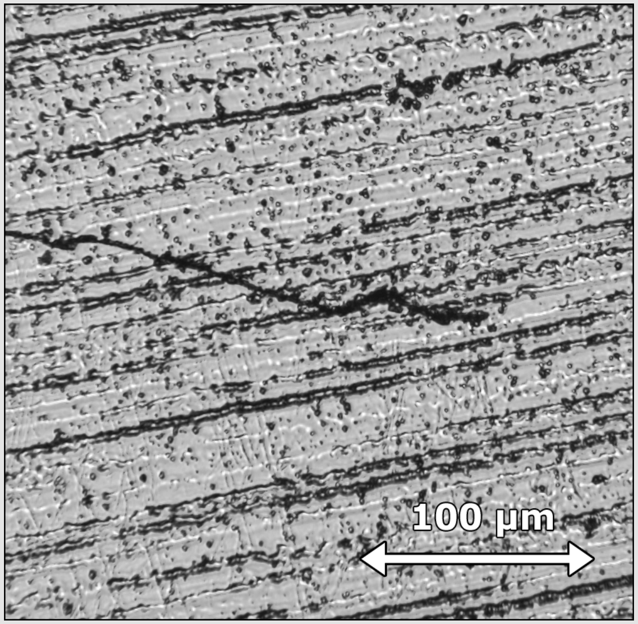


Alloy 152: Constant K

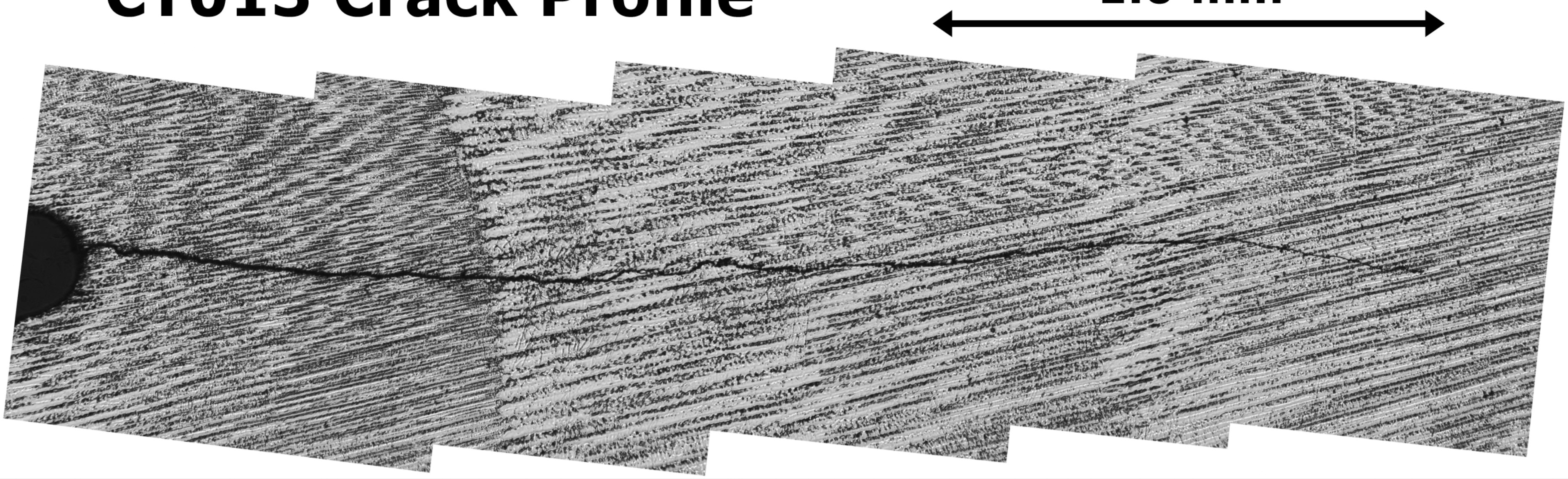


Post-Test Crack Profile

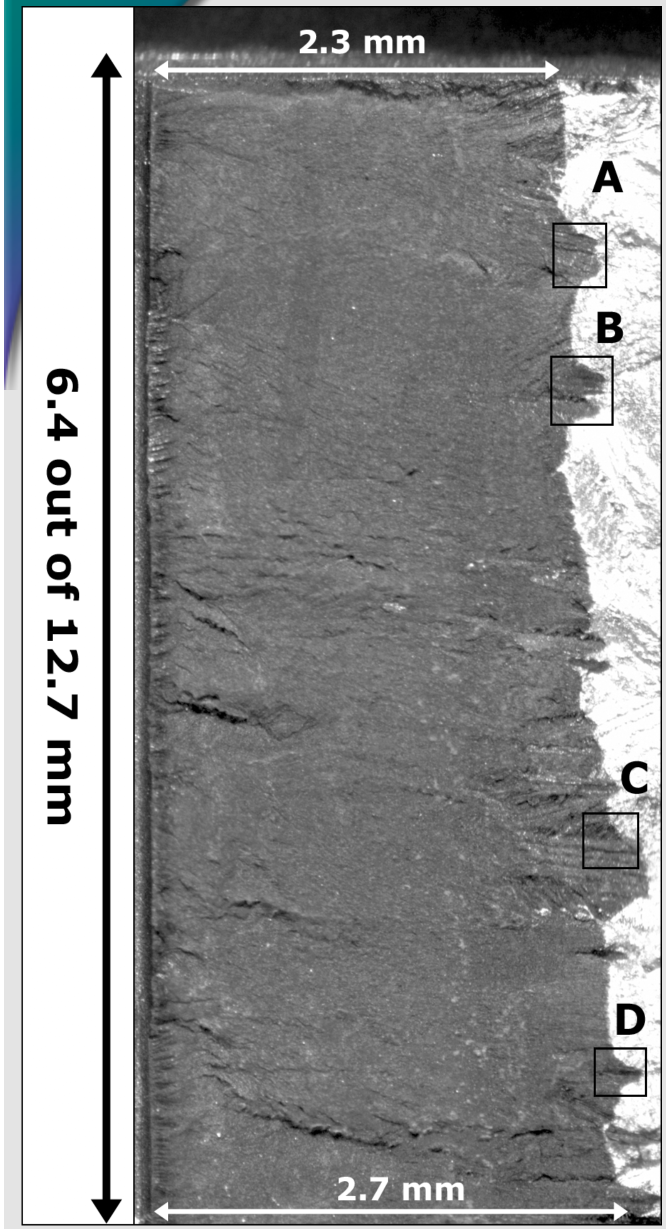
- *As planned, precracked into single weld pass for SCC test.*
- *Crack growth during cycling does not follow dendrite structure or grain boundaries.*
- *IGSCC limited to last few μm of crack growth during hold times and constant K.*



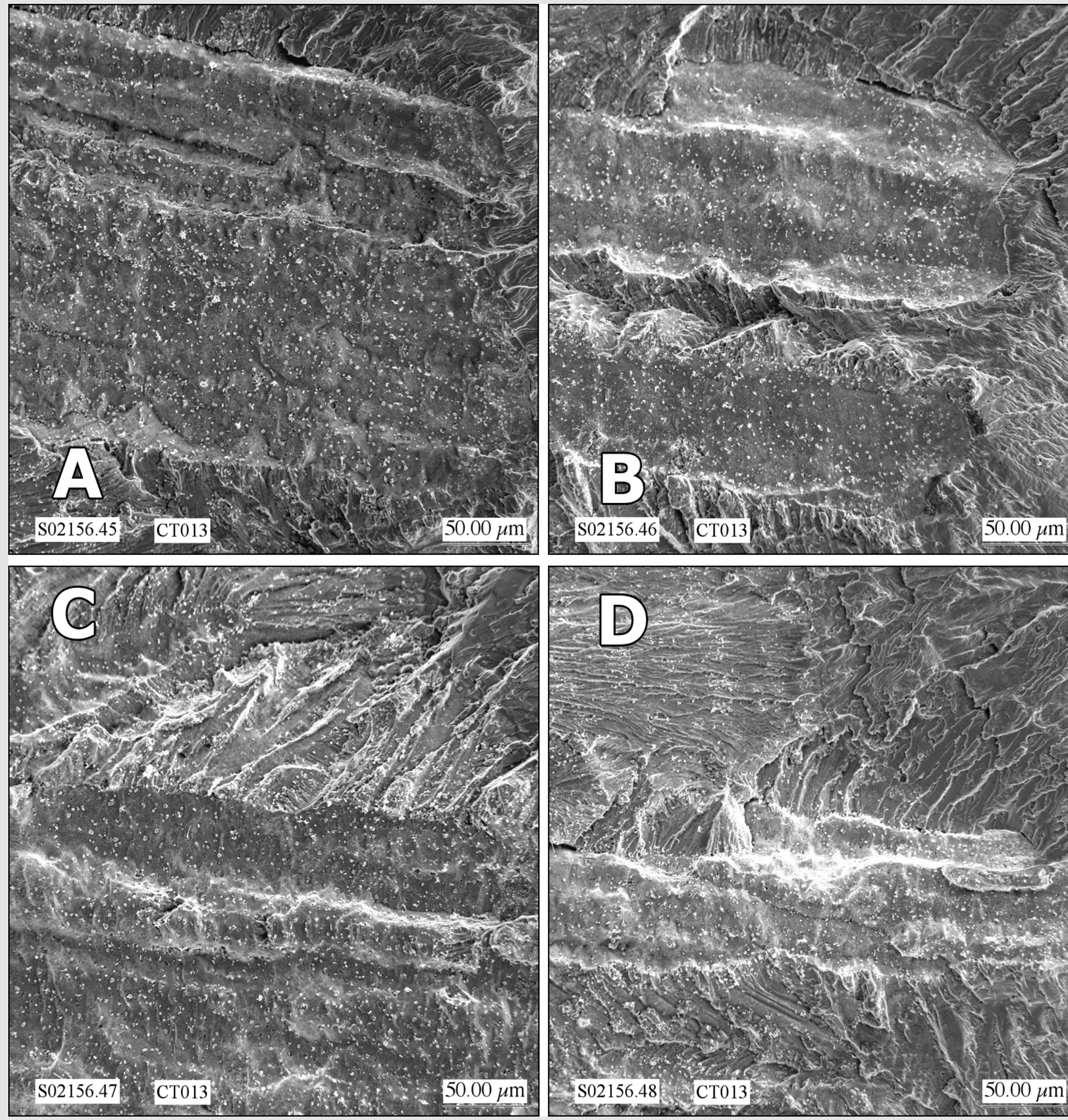
CT013 Crack Profile



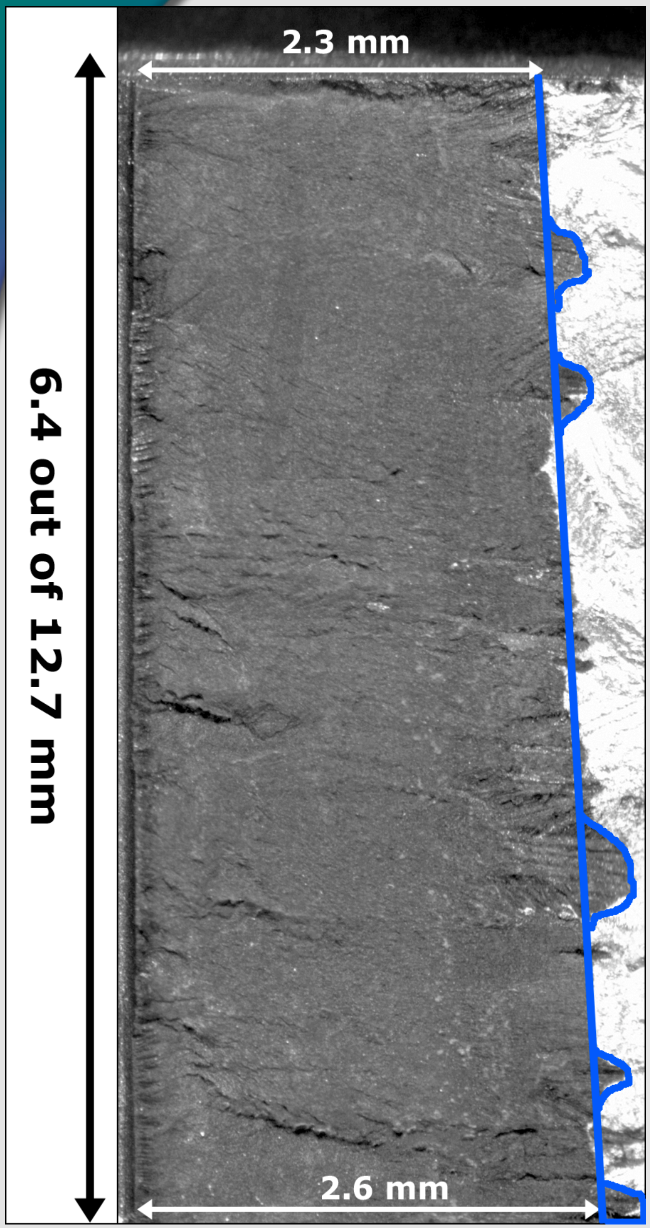
Post-Test Fracture Surface



Intergranular SCC seen at crack front



SCC CGRs from Crack Surface



Estimate of CGR based on crack front extensions

- *Assume SCC crack growth limited to local regions extending beyond straight line drawn across crack front.*
- *Calculate area of extensions and divide by width of specimen section which gives an "average" SCC crack extension across the specimen thickness.*
- *Assuming that SCC growth begins at onset of first step with the 2.5-h hold time (at 800 h), can obtain an estimated correction factor for the DCPD values.*
- *Results in a SCC growth rate at constant K of ~$3x10^{-10}$ mm/s.*

Alloy 152
Test #1 Summary

- *Alloy 152 weld metal was resistant to SCC in simulated primary PWR water at 325°C even when the pre-crack is oriented along dendrite boundaries in a single pass.*

- *DCPD indicated stable crack advance under gentle cycle + hold conditions and constant K conditions, but at an extremely low propagation rates.*

- *Fractography reveals IGSCC during the cycle + hold and constant K loading conditions. "Corrected" CGR under constant K is approximately $3x10^{-10}$ mm/s.*

PNNL Test Status: October 2007

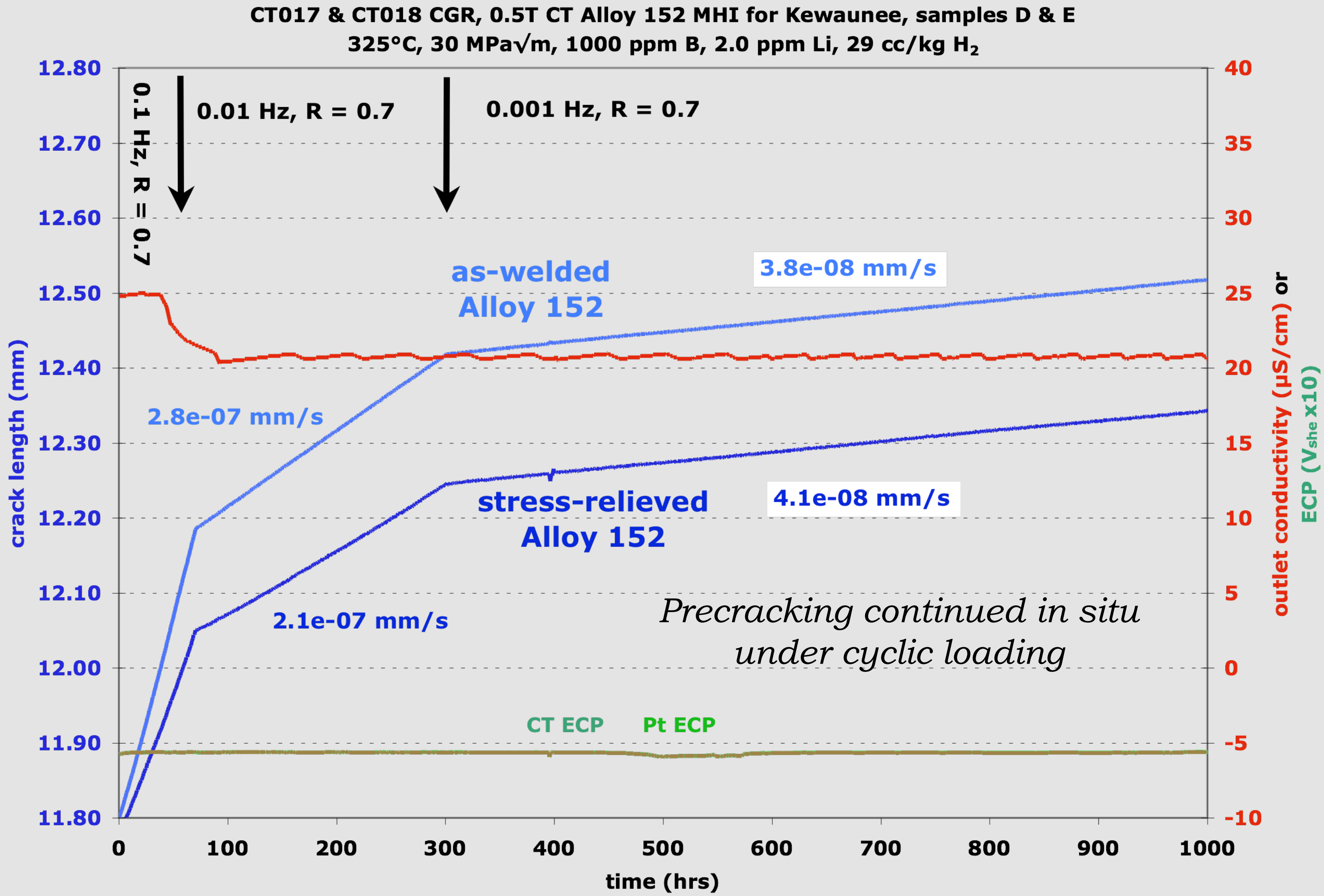
Alloy 152

- Alloy 152 Weld Metal in 325°C PWR primary water
- ***Alloy 152 Weld Metal in 325-350°C PWR primary water; as welded and stress relieved conditions***

Alloy 690

- Alloy 690 CRDM heat RE243 in 325°C PWR primary water; as-received TT and SA conditions
- Alloy 690 CRDM heat RE243 in 325-350°C PWR primary water; as-received TT and SA conditions; 1D cold rolled to 17% reduction in SL orientation and 1D cold rolled to 30% reduction in TL orientation
- Alloy 690 CRDM heats RE076, WP140 and WP142 in 350°C PWR primary water; as-received TT condition

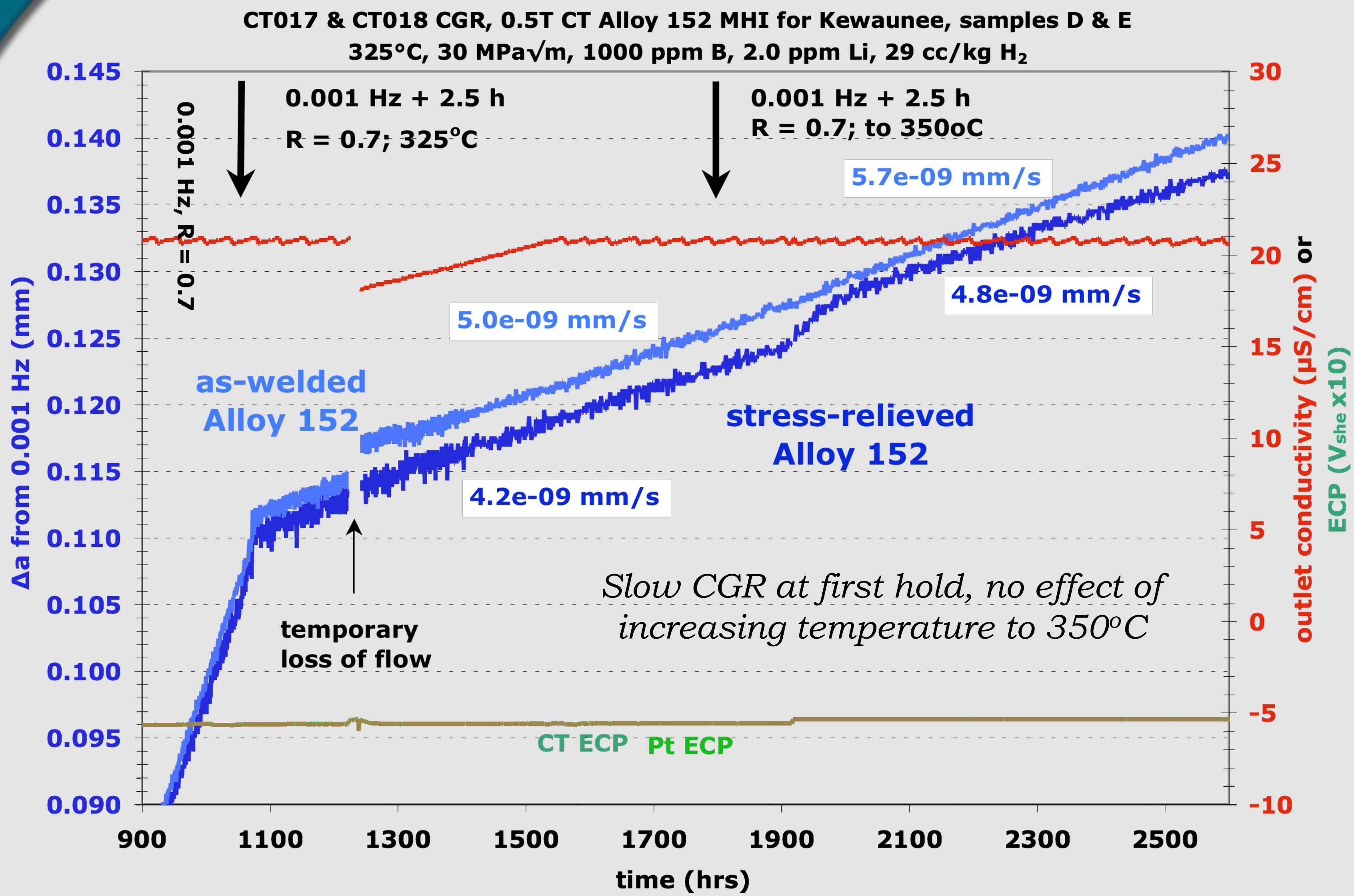
Alloy 152 (AW/SR) Precracking and Transitioning



Battelle

Pacific Northwest National Laboratory

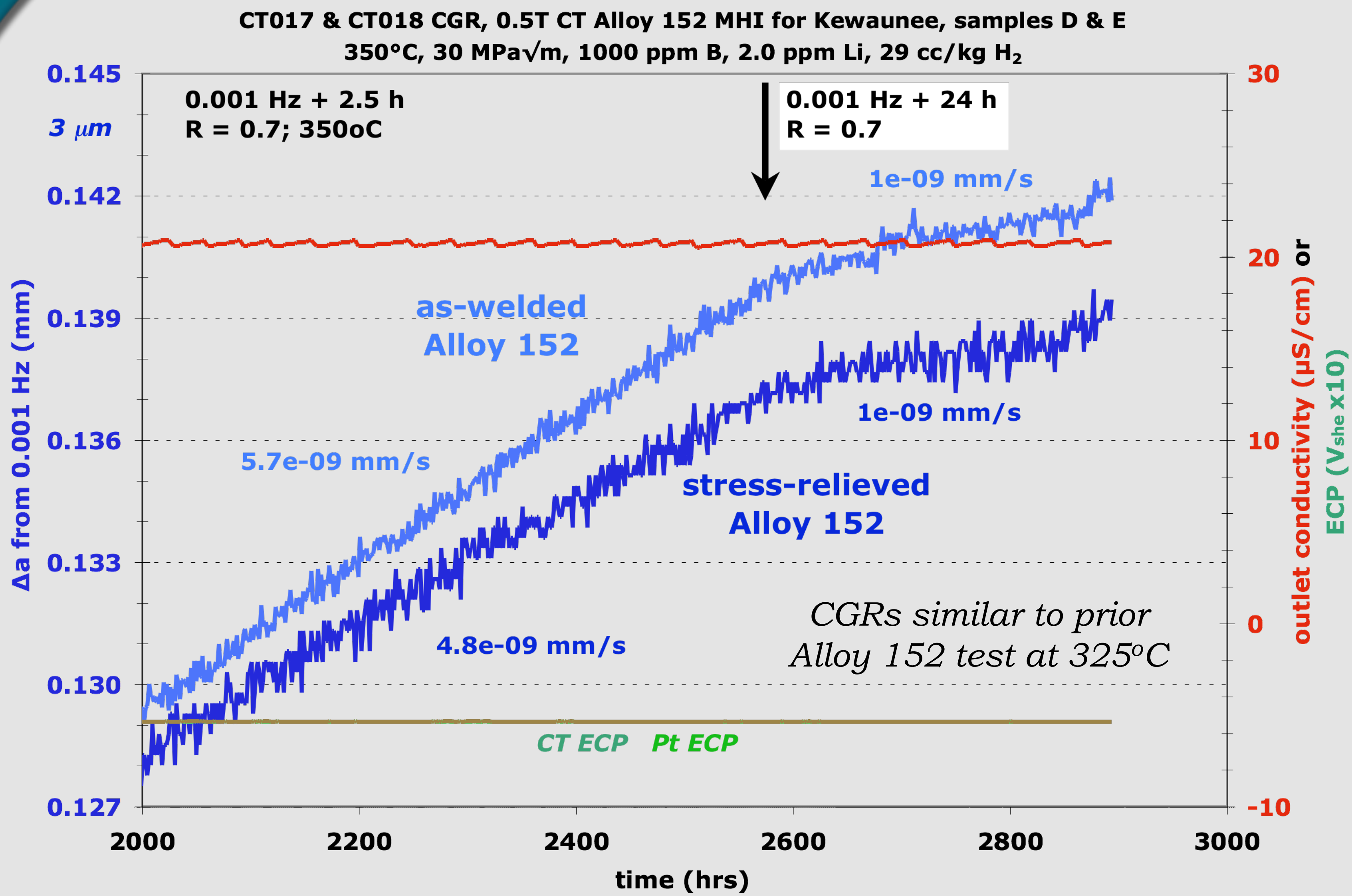
Alloy 152 (AW/SR): Cycle + Hold



Battelle

Pacific Northwest
National Laboratory

Alloy 152 (AW/SR): Cycle + Hold



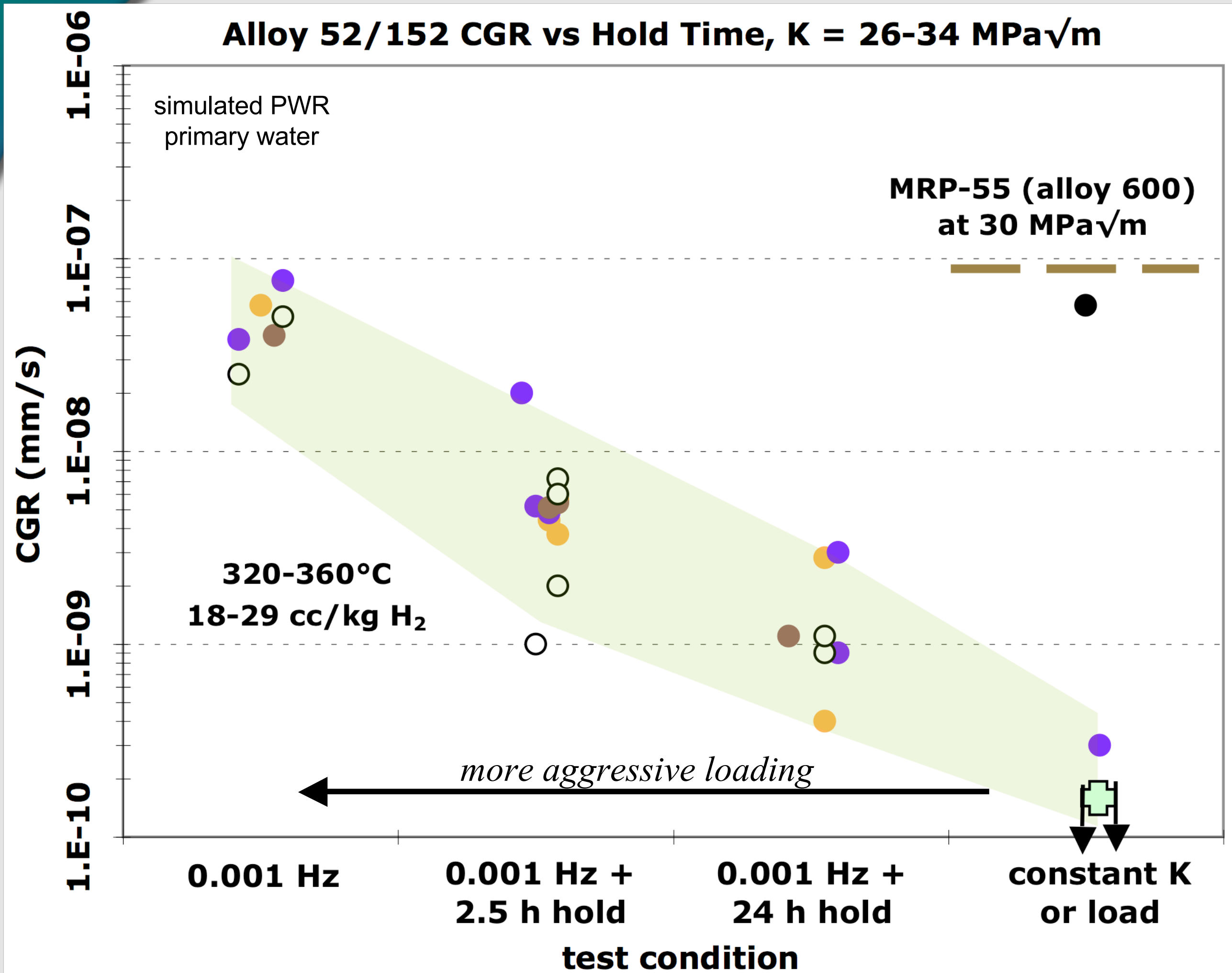
Alloy 152 CGR Data Summary as of 10/07

specimen ID	heat	TMT	water chem	DH (cc/kg)	test temp (C)	K (MPa√m)	load ratio	loading conditions	Cond. #	crack ext incr (μm)	time (h)	CGR (mm/s)
CT013	EPRI -MHI	as-welded	1000B, 2.0Li	29	325	30	0.7	0.001 Hz	1	100	600	7.7×10^{-08}
complete			1000B, 2.0Li	29	325	32	0.7	0.001 Hz + 2.5 h	2	10	500	2.0×10^{-08}
Corrected K & CGR			1000B, 2.0Li	29	325	33	0.7	0.001 Hz + 24 h	3	3.5	800	2.2×10^{-09}
			1000B, 2.0Li	29	325	33	1.0	constant K	4	0.5	2000	3.0×10^{-10}
CT017	EPRI -MHI	stress-relieved	1000B, 2.0Li	29	325	30	0.7	0.001 Hz	1	100	750	4.1×10^{-08}
in progress			1000B, 2.0Li	29	325	30	0.7	0.001 Hz + 2.5 h	2	15	850	5.0×10^{-09}
dcpd only			1000B, 2.0Li	29	350	30	0.7	0.001 Hz + 2.5 h	2	6	350	5.7×10^{-09}
			1000B, 2.0Li	29	350	30	0.7	0.001 Hz + 24 h	3	2	350	1.0×10^{-09}
CT018	EPRI -MHI	as-welded	1000B, 2.0Li	29	325	30	0.7	0.001 Hz	1	100	750	3.8×10^{-08}
in progress			1000B, 2.0Li	29	325	30	0.7	0.001 Hz + 2.5 h	2	15	850	4.2×10^{-09}
dcpd only			1000B, 2.0Li	29	350	30	0.7	0.001 Hz + 2.5 h	2	6	350	4.8×10^{-09}
			1000B, 2.0Li	29	350	30	0.7	0.001 Hz + 24 h	3	2	350	1.0×10^{-09}

Alloy 152 Summary

- *Initial tests show that alloy 152 weld metal is resistant to SCC propagation in simulated primary PWR water at 325-350°C even when the pre-crack is oriented along dendrite boundaries in a single pass.*
- *DCPD indicated stable crack advance under gentle cycle + hold conditions and constant K conditions, but at an extremely low propagation rates.*
- *Fractography reveals IGSCC during the cycle + held and constant K loading conditions in the first test.*
- *Tests on two alloy 152 samples (as welded and stress relieved) are continuing to constant K. So far consistent SCC response with initial test.*

Alloy 152/52 CGR Data Assessment



SCC response can also be evaluated during cycle + hold loading to expand very limited data base. Improves confidence in constant K SCC response.

- *Extremely limited SCC-CGR data for alloy 152 or alloy 52 weld metal.*
- *Consistent, extremely low SCC-CGR response for GEG/PNNL welds, ANL result suggests possible important weld-to-weld variability?*

PNNL Test Status: October 2007

Alloy 152

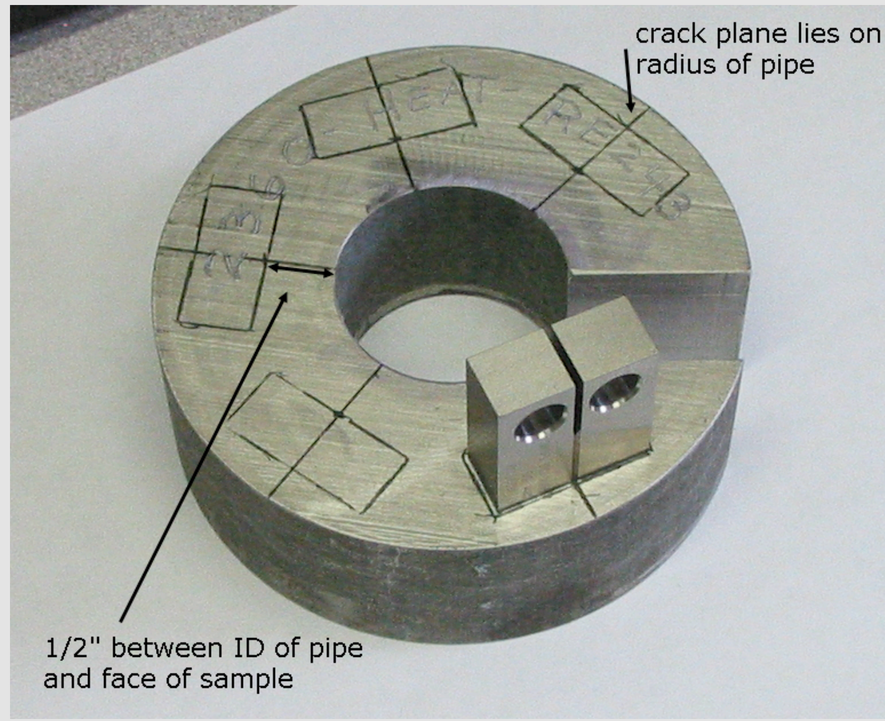
- Alloy 152 Weld Metal in 325°C PWR primary water
- Alloy 152 Weld Metal in 325-350°C PWR primary water; as welded and stress relieved conditions

Alloy 690

- ***Alloy 690 CRDM heat RE243 in 325°C PWR primary water; as-received TT and SA conditions***
- Alloy 690 CRDM heat RE243 in 325-350°C PWR primary water; as-received TT and SA conditions; 1D cold rolled to 17% reduction in SL orientation and 1D cold rolled to 30% reduction in TL orientation
- Alloy 690 CRDM heats RE076, WP140 and WP142 in 350°C PWR primary water; as-received TT condition

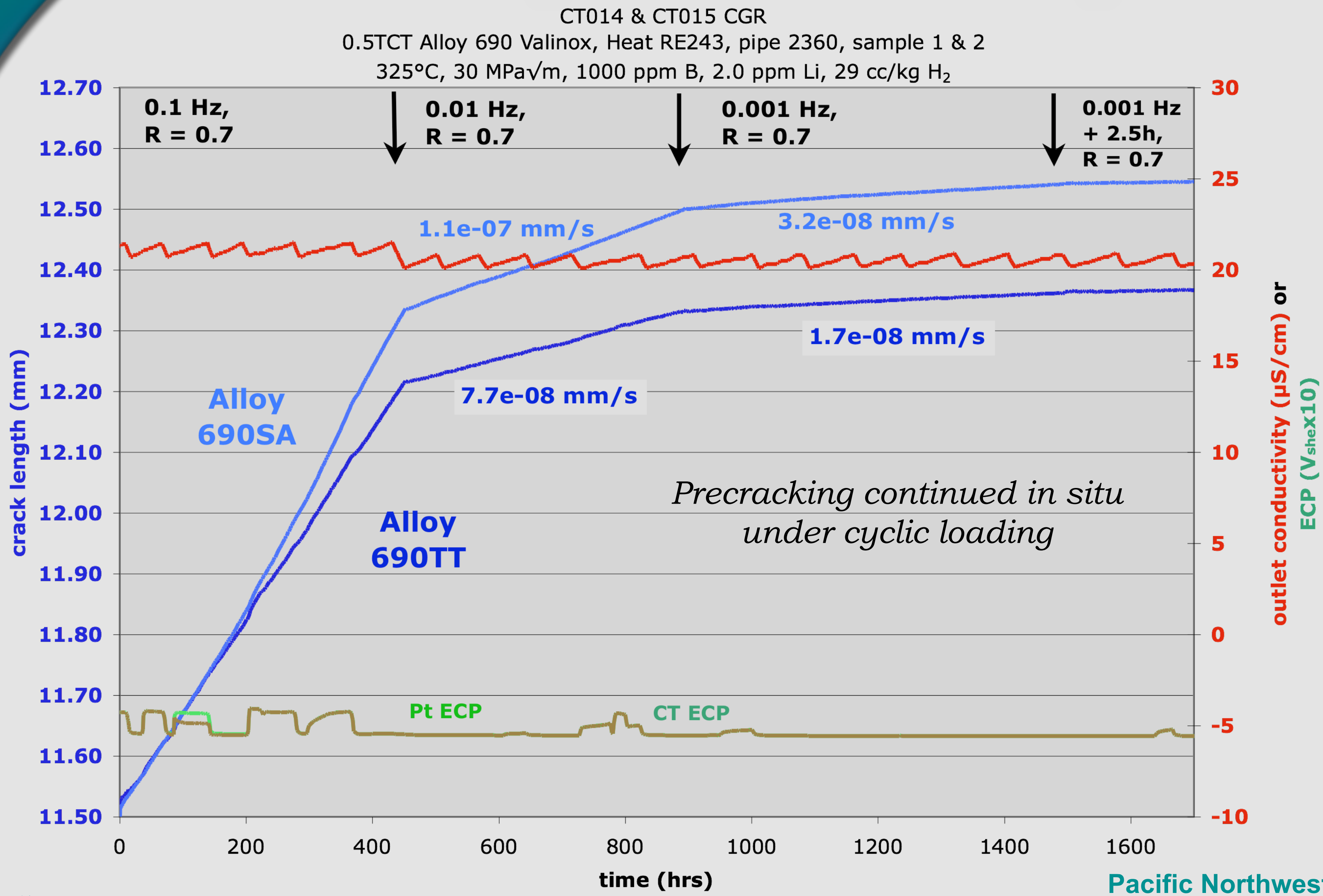
Alloy 690 CRDM Materials

Six different heats with tubing wall thicknesses from 25 to 36 mm obtained from Valinox Nucleaire and one 34-mm-thick plate from EPRI.



Initial testing on alloy 690 tube heat RE243; CT section blanks sectioned as shown in the C-L orientation. Material was thermally treated in the as-received condition, selected samples solution annealed (1100°C/1 h and water quenched) and others cold rolled in S-L or T-L orientations before machining into 0.5T CTs.

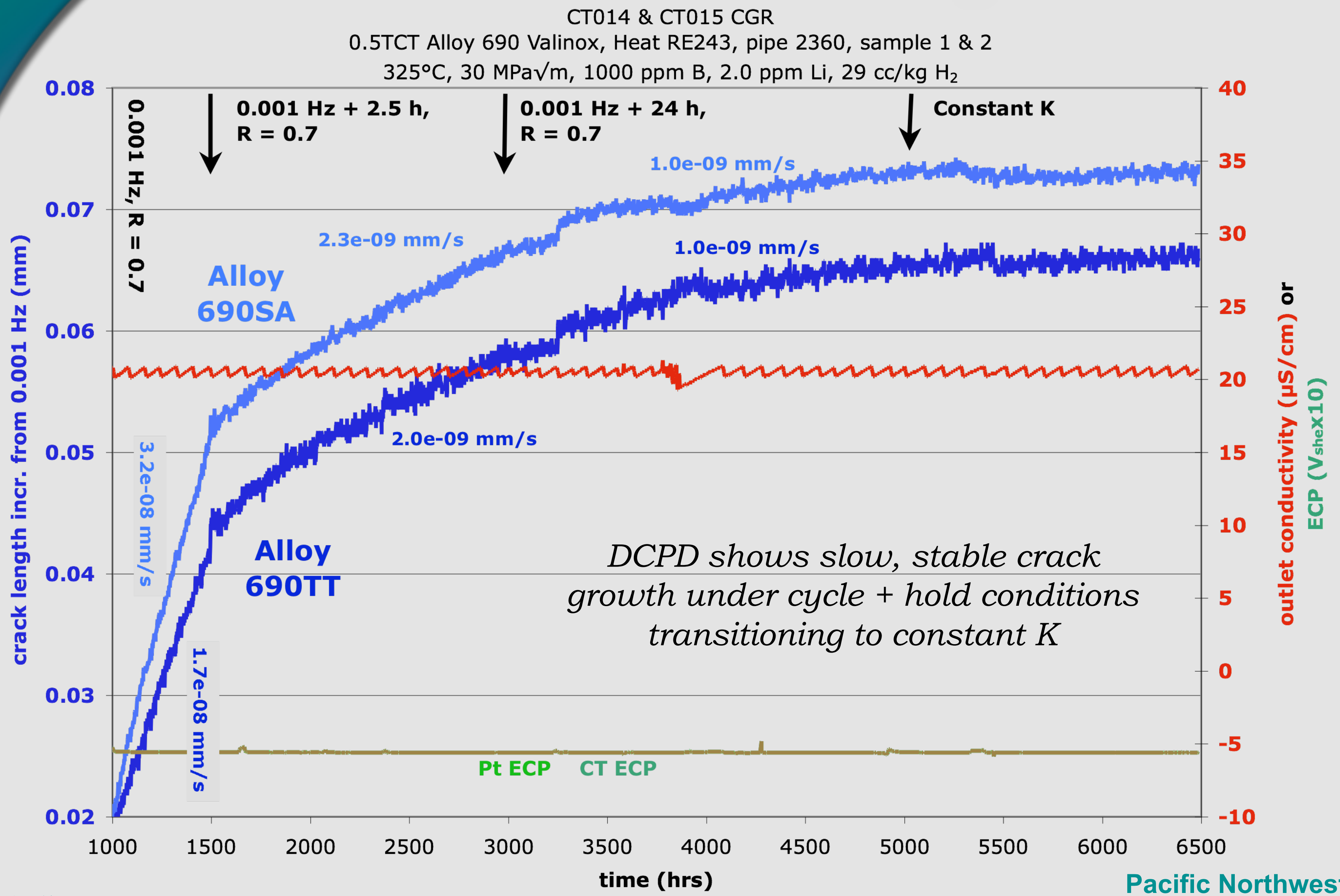
Alloy 690 (TT/SA) Precracking and Transitioning



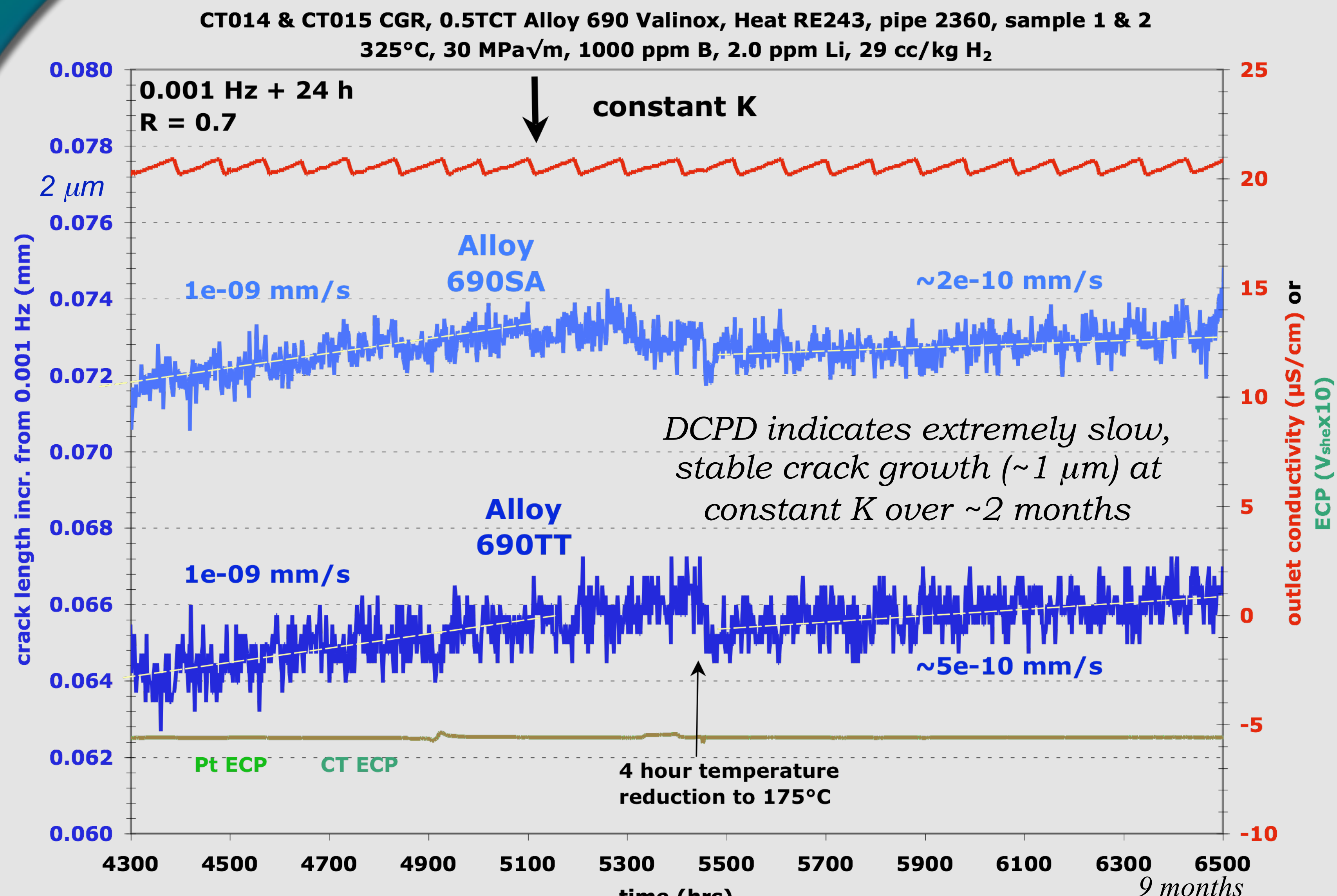
Battelle

Pacific Northwest National Laboratory

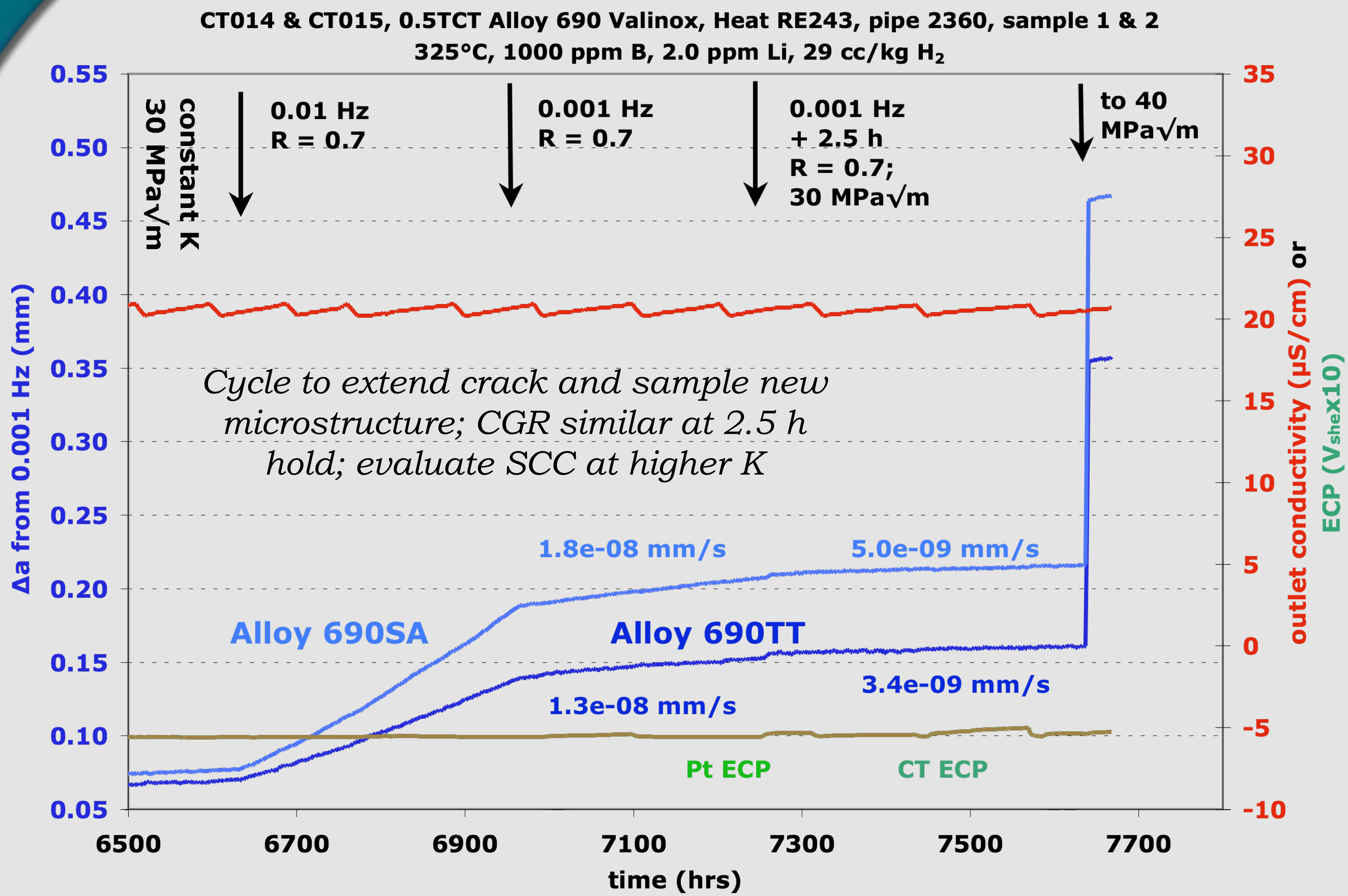
Alloy 690 (TT/SA) Crack Transitioning



Alloy 690 (TT/SA): Constant K



Continue Alloy 690 (TT/SA): Increase K



Alloy 690 CGR Data Summary as of 10/07

spec ID	heat	TMT	water chem	DH cc/kg	test temp (C)	K MPa√m	load ratio	loading conditions	Cond #	crack ext incr (µm)	time (h)	CGR (mm/s)
CT014	RE243 pipe 2360	AR(TT)	1000B, 2.0Li	29	325	30	0.7	0.001 Hz	1	30	600	$1.0\text{x}10^{-08}$
in progress			1000B, 2.0Li	29	325	30	0.7	0.001 Hz + 2.5 h	2	14	1500	$6.8\text{x}10^{-09}$
dcpd only			1000B, 2.0Li	29	325	30	0.7	0.001 Hz + 24 h	3	5	2000	$1.0\text{x}10^{-09}$
			1000B, 2.0Li	29	325	30	1.0	constant K	4	1.5	1400	$<5\text{x}10^{-10}$
			1000B, 2.0Li	29	325	30	0.7	0.001 Hz	1	12	350	$1.3\text{x}10^{-08}$
			1000B, 2.0Li	29	325	30	0.7	0.001 Hz + 2.5 h	2	4	350	$3.4\text{x}10^{-09}$
			1000B, 2.0Li	29	325	40	0.7	0.001 Hz + 2.5 h	2	2	150	$\sim 4\text{x}10^{-09}$
CT015	RE243, pipe 2360	AR+SA 1100C/1h	1000B, 2.0Li	29	325	30	0.7	0.001 Hz	1	40	600	$1.6\text{x}10^{-08}$
in progress			1000B, 2.0Li	29	325	30	0.7	0.001 Hz + 2.5 h	2	15	1500	$3.2\text{x}10^{-09}$
dcpd only			1000B, 2.0Li	29	325	30	0.7	0.001 Hz + 24 h	3	5	2000	$1.0\text{x}10^{-09}$
			1000B, 2.0Li	29	325	30	1.0	constant K	4	0.7	1400	$<2\text{x}10^{-10}$
			1000B, 2.0Li	29	325	30	0.7	0.001 Hz	1	20	350	$1.8\text{x}10^{-08}$
			1000B, 2.0Li	29	325	40	0.7	0.001 Hz + 2.5 h	2	2	150	$\sim 4\text{x}10^{-09}$

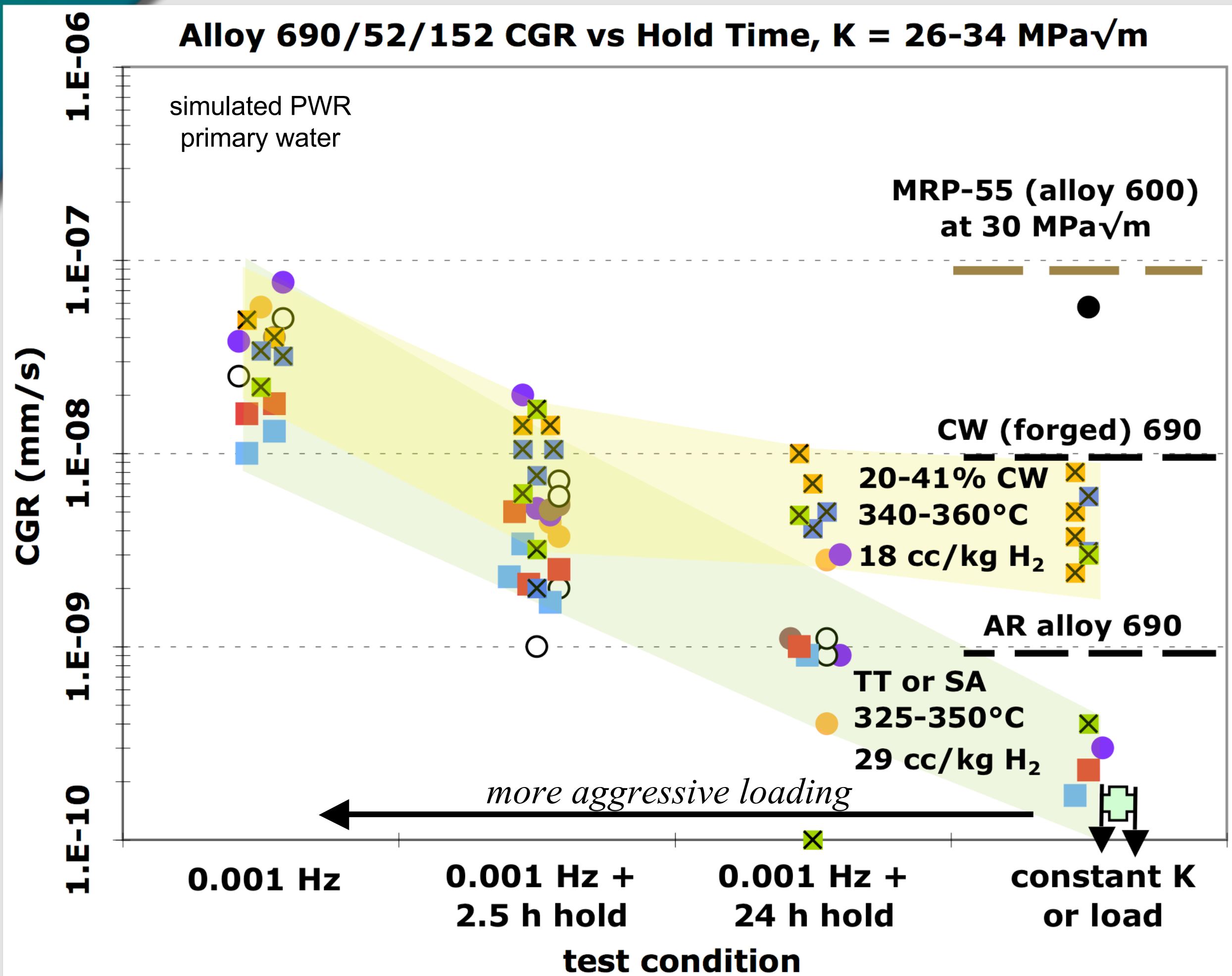
Alloy 690 Summary

- *Initial tests show that alloy 690 CRDM tubing is resistant to SCC propagation in simulated primary PWR water at 325°C with similar response for the as-received, TT and carbide-modified SA conditions.*

- *DCPD indicated stable crack advance under gentle cycle + hold and constant K conditions, but at an extremely low propagation rates. DCPD-measured CGRs under constant K were <5x10^{-10} mm/s.*

- *This test is continuing to briefly evaluate SCC response at a higher K level. As a result, fractography has not been performed to correct CGRs or establish the morphology of SCC.*

Alloy 690/152/52 CGR Data

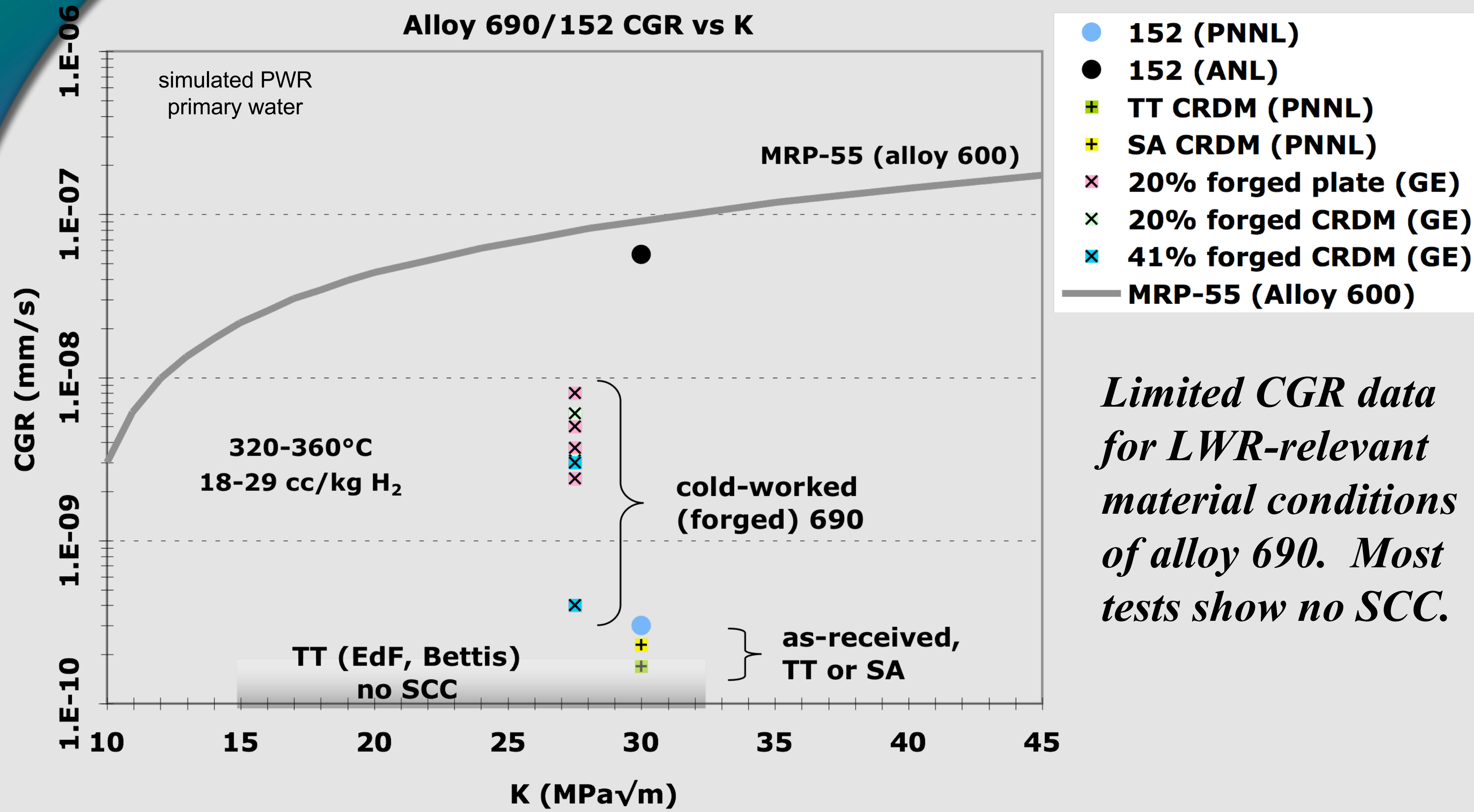


SCC response can also be evaluated during cycle + hold loading to expand very limited data base.

Alloy 152 data generally consistent with alloy 690.

- *Cold worked alloy 690 has higher CGRs across range of conditions.*
- *Low CGRs even with "gentle" cycling, helps establish confidence for low constant K SCC CGRs of <10^{-9} mm/s AR and <10^{-8} mm/s CW.*

Alloy 690/152 Data (Excluding 1D Rolled)



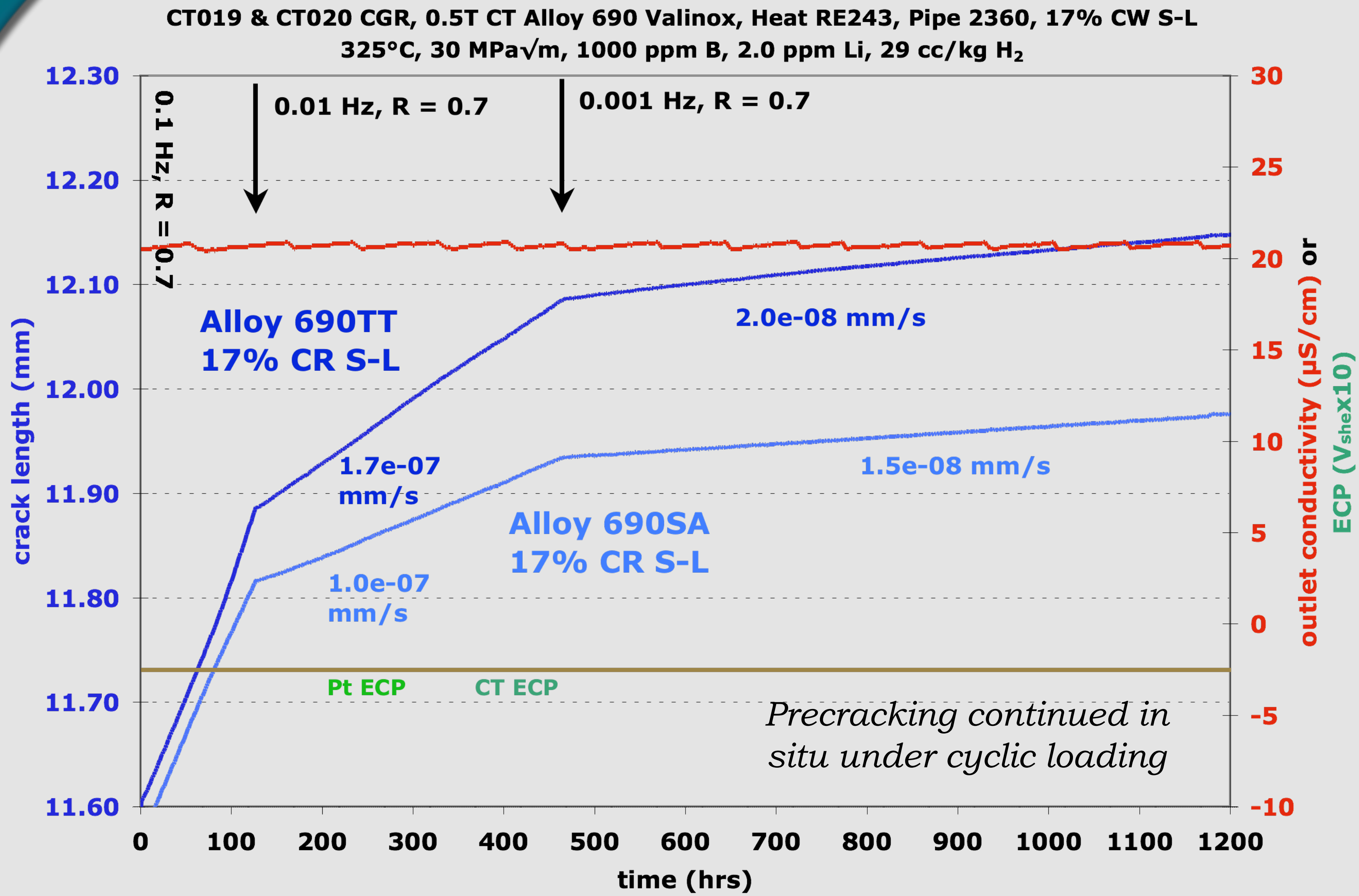
Limited CGR data for LWR-relevant material conditions of alloy 690. Most tests show no SCC.

- *As-received, thermally treated or solution annealed alloy 690 exhibits extremely low constant K, CGRs (<$1x10^{-9}$ mm/s) or no SCC.*
- *Cold work by forging increases CGRs, still <$1x10^{-8}$ mm/s.*

PNNL Test Status: October 2007

- **Alloy 152**
  - Alloy 152 Weld Metal in 325°C PWR primary water
  - Alloy 152 Weld Metal in 325-350°C PWR primary water; as welded and stress relieved conditions
- **Alloy 690**
  - Alloy 690 CRDM heat RE243 in 325°C PWR primary water; as-received TT and SA conditions
  - ***Alloy 690 CRDM heat RE243 in 325-350°C PWR primary water; as-received TT and SA conditions; 1D cold rolled to 17% reduction in S-L orientation*** and 1D cold rolled to 17-30% reduction in T-L orientation
  - Alloy 690 CRDM heats RE076, WP140 and WP142 in 350°C PWR primary water; as-received TT condition

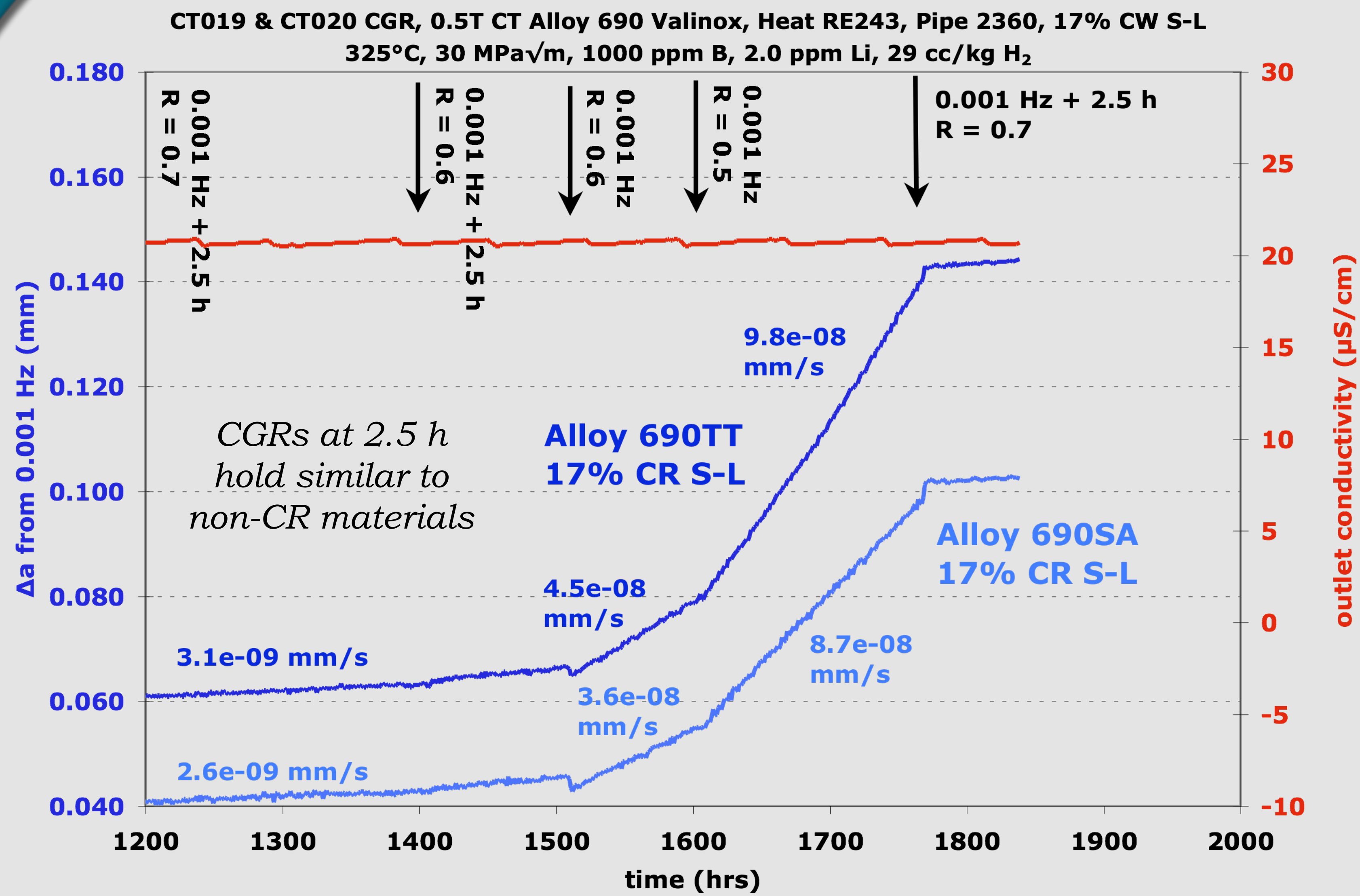
Alloy 690 (1D - SL) Precracking and Transitioning



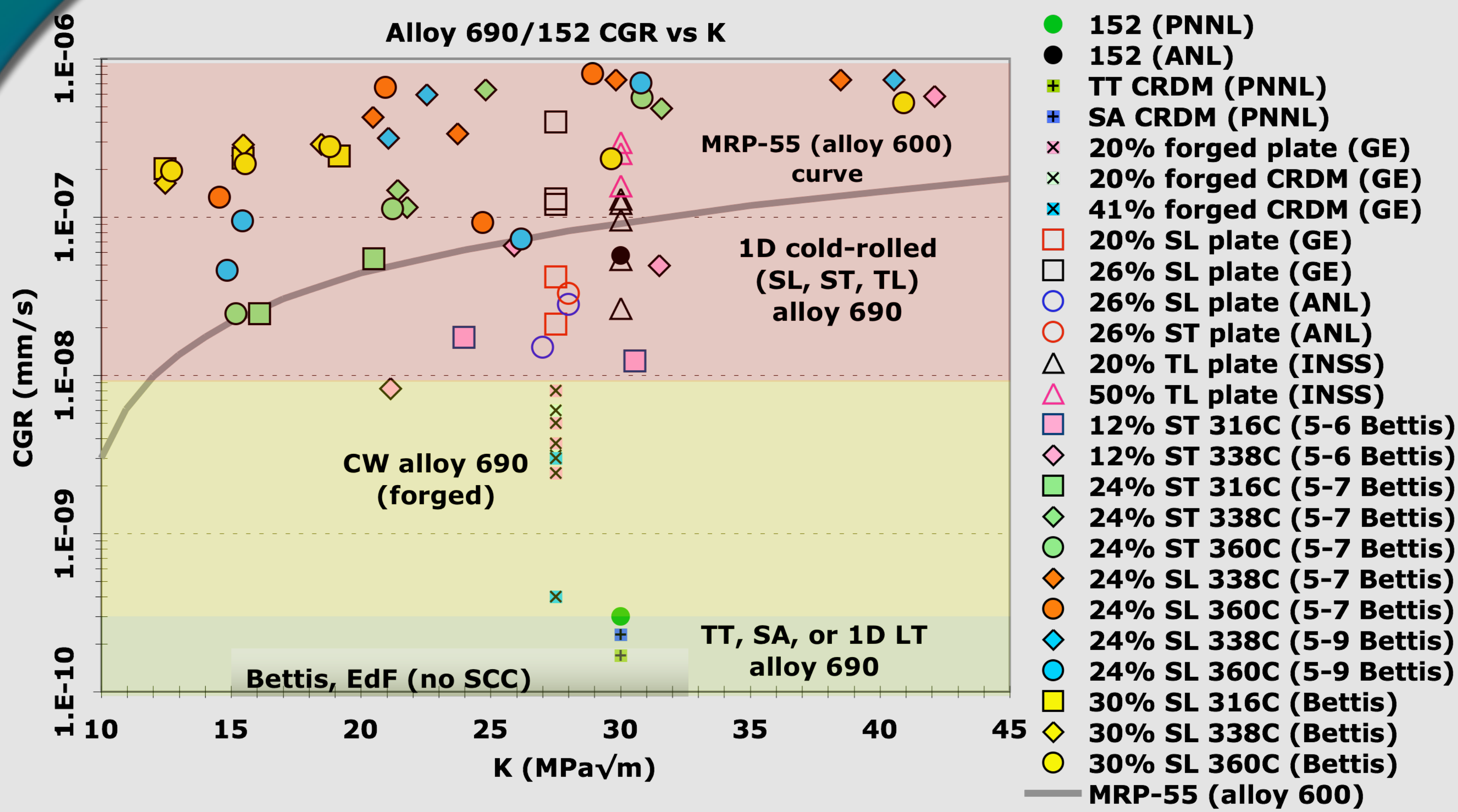
Battelle

Pacific Northwest
National Laboratory

Alloy 690 (1D - SL) Crack Transitioning



All Alloy 690/152/52 - K Dependence



- *1D cold-rolled (S-L, S-T, T-L) materials exhibit high CGRs at constant K or constant load even at lower K values.*
- *Are 1D rolled materials relevant for LWR service components? Must understand high SCC susceptibility.*

PNNL Near-Term SCC-CGR Test Plans

➢ Alloy 152 Weldments

- Complete tests on alloy 152 weld metal in 350°C PWR primary water for as welded and stress relieved conditions.
- Begin tests evaluating regions near the alloy 152 - SS fusion zone within the dilution zone.

➢ Alloy 690 CRDM Tubing Heats

- Complete tests on alloy 690 CRDM heat RE243 in 325°C PWR primary water for as-received TT and SA conditions.
- Complete tests on alloy 690 CRDM heat RE243 in 325-350°C PWR primary water for as-received TT and SA conditions and 1D cold rolled (CR) to 17% reduction in SL orientation.
- Begin tests on alloy 690 heat RE243 in as-received TT condition and CR to 17% & 30% reduction in TL orientation.
- Begin tests on alloy 690 heats RE076, WP140 and WP142 in 350°C PWR primary water on as-received TT condition.

Tests are also being performed on alloy 600/182 service and lab materials.

Major Task Areas

- **Crack-Growth System Construction/Testing**
  - CGR systems and validation testing completed in 2006.
- **Crack-Growth Rate Testing**
  - Austenitic Stainless Steels
  - Alloy 600 and Alloy 182 Weld Metal
  - Alloy 690 and Alloy 152/52 Weld Metals
- **Characterization and Cracking Mechanisms**
  - OM, SEM, EBSD and TEM examinations of test materials
  - SEM/ATEM characterizations of SCC cracks and crack tips in LWR service materials and laboratory CGR samples
  - SEM/ATEM characterizations of hot cracks in mockup and laboratory alloy 182/82 and alloy 152/52 weldments

Characterization and Cracking Mechanisms

Contributions from Matt Olszta and Larry Thomas

➢ OM, SEM, EBSD and TEM Examinations of CGR Test Materials

- *Establish heat-to-heat microstructural variability in alloy 690 product forms and in alloy 152/52 welds.*
- *Investigate processing, heat treatment and cold work effects on matrix and grain boundary microstructures.*

➢ SEM/ATEM Characterizations of SCC Cracks and Crack Tips

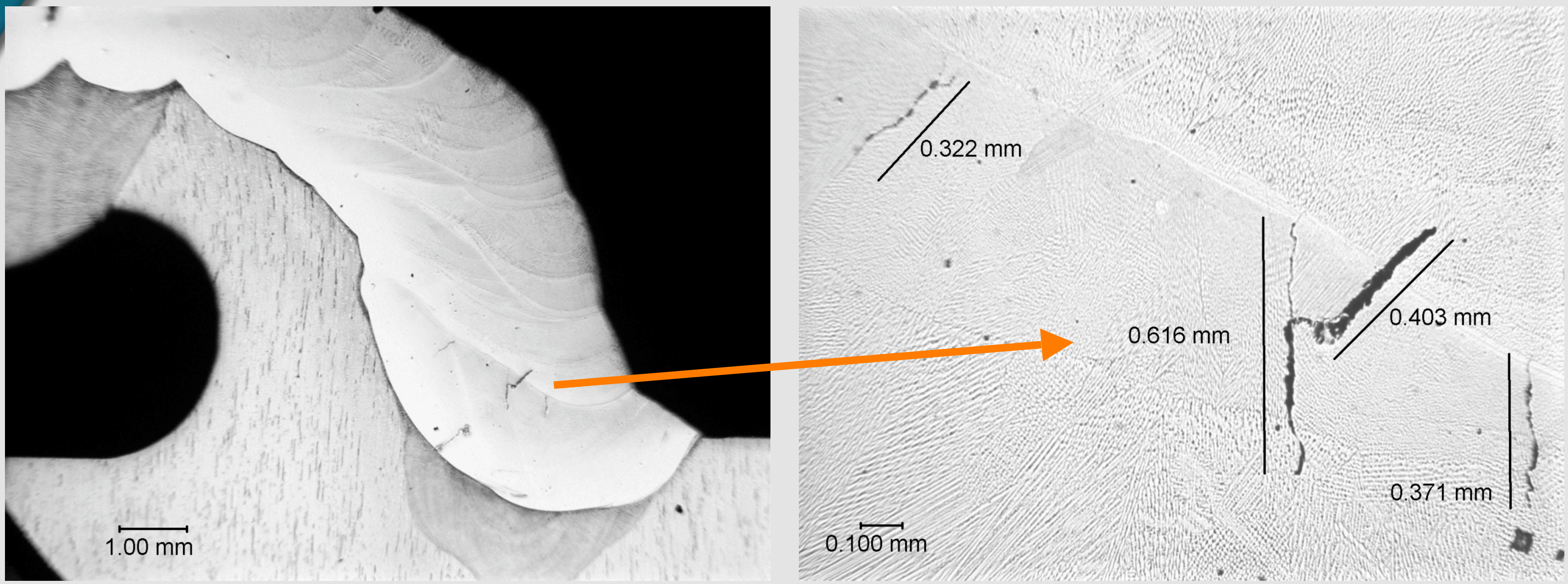
- *Examine H_2 & ECP effects on SCC propagation mechanisms in 300-series stainless steel and alloy 600/182.*
- *Evaluate crack tip and SCC mechanism differences for alloy 600/690 and alloy 182/152.*

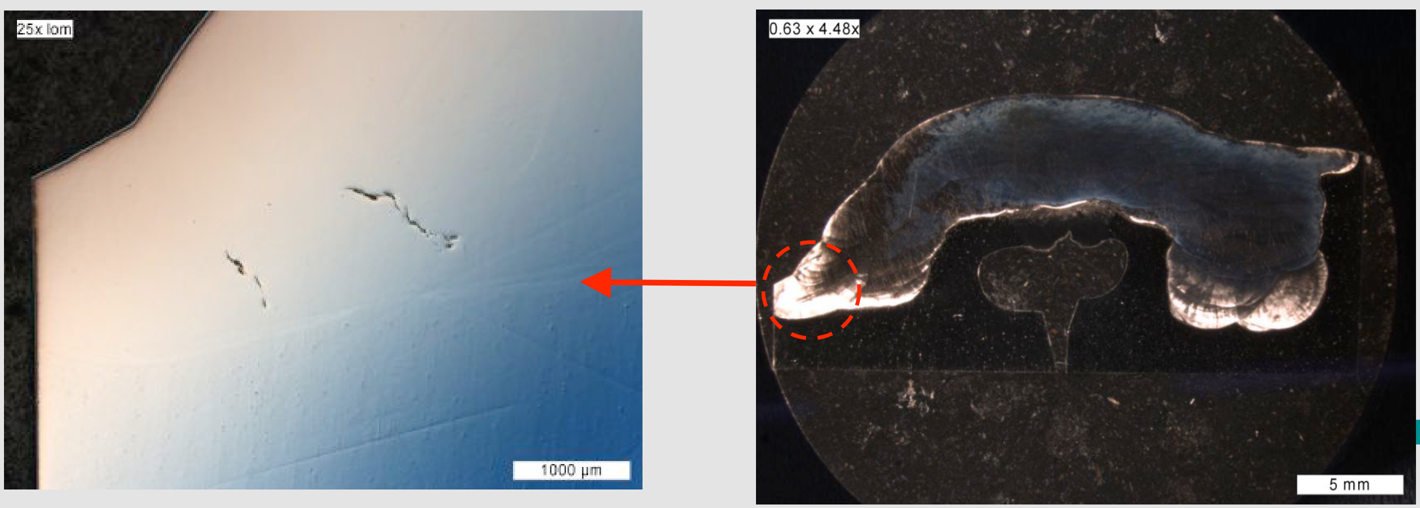
➢ SEM/ATEM Characterizations of Hot Cracks

- *Investigate hot cracking mechanisms in field mockup welds and laboratory test welds for alloy 152/52 and 182/82 weldments.*

Weld Overlay Repair Mockup using Alloy 52 on Stainless Steel: Hot Cracks in Dilution Zone

from Pål Efsing and Björn Forssgren, Ringhals AB





Alloy 52 Overlay Weld Samples

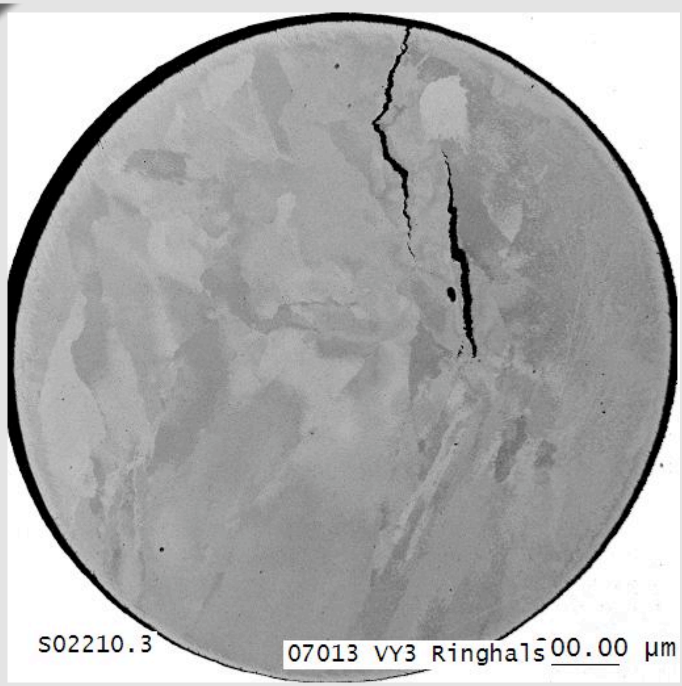
from
Pål Efsing
Ringhals AB

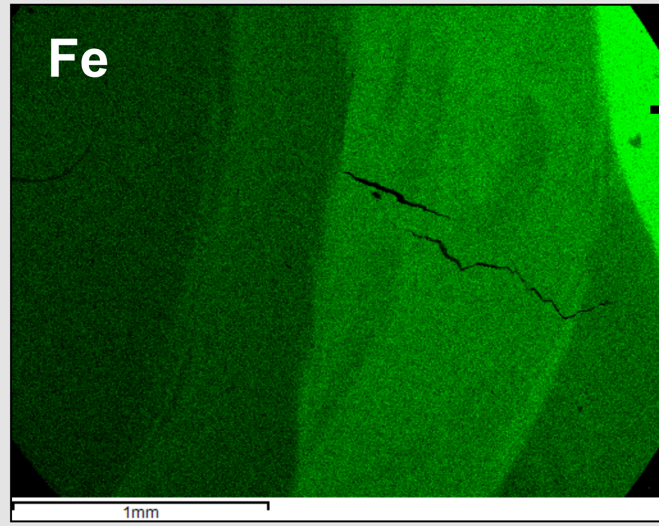
Vy4

Initial TEM Sample Locations for Hot Cracks

Vy3

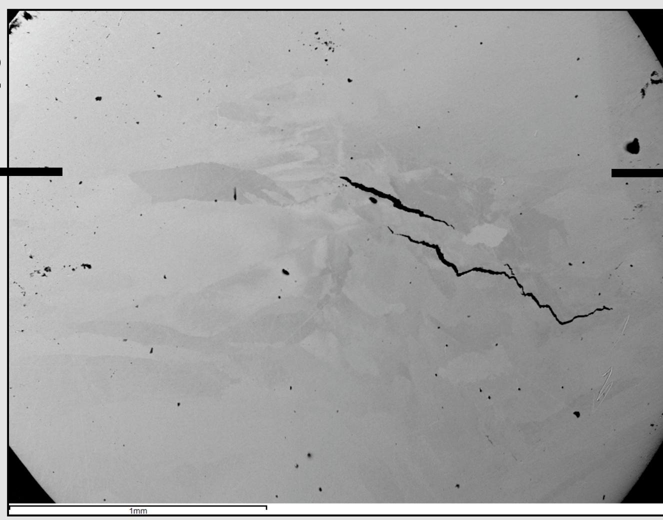
Alloy 52 Overlay Weld Sample VY3



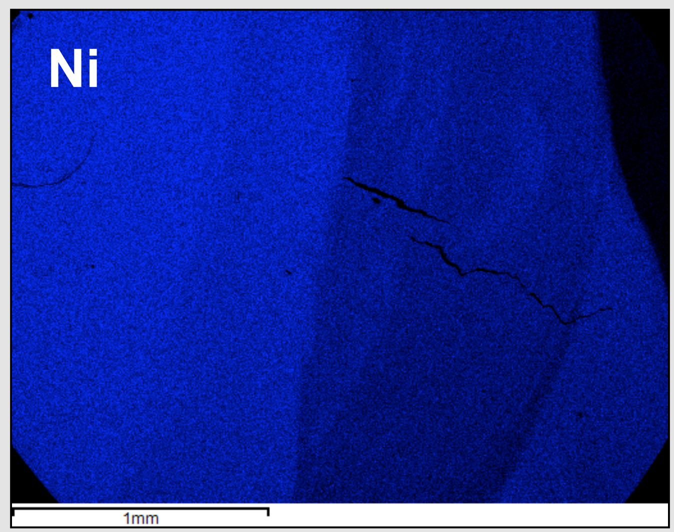


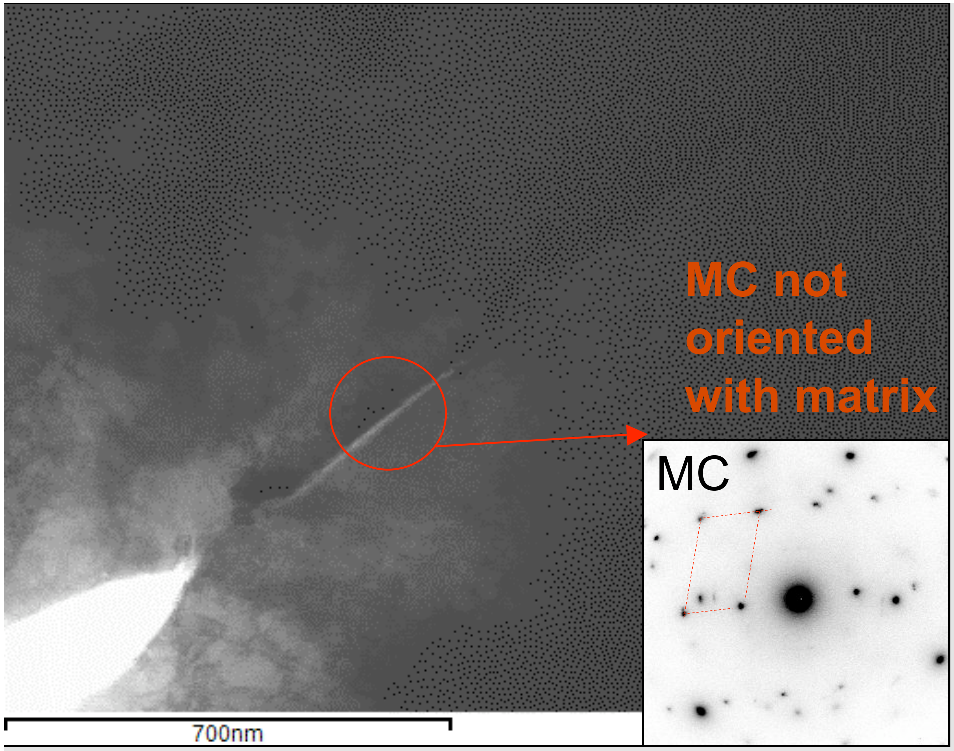
SS

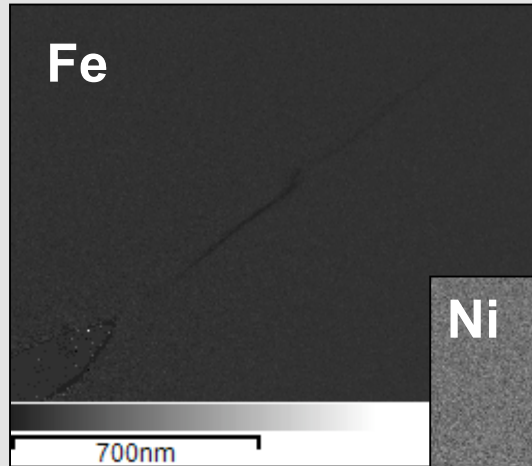
Alloy 52
Weld



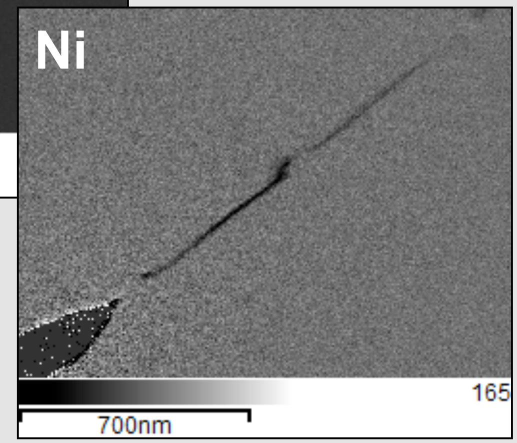
SS

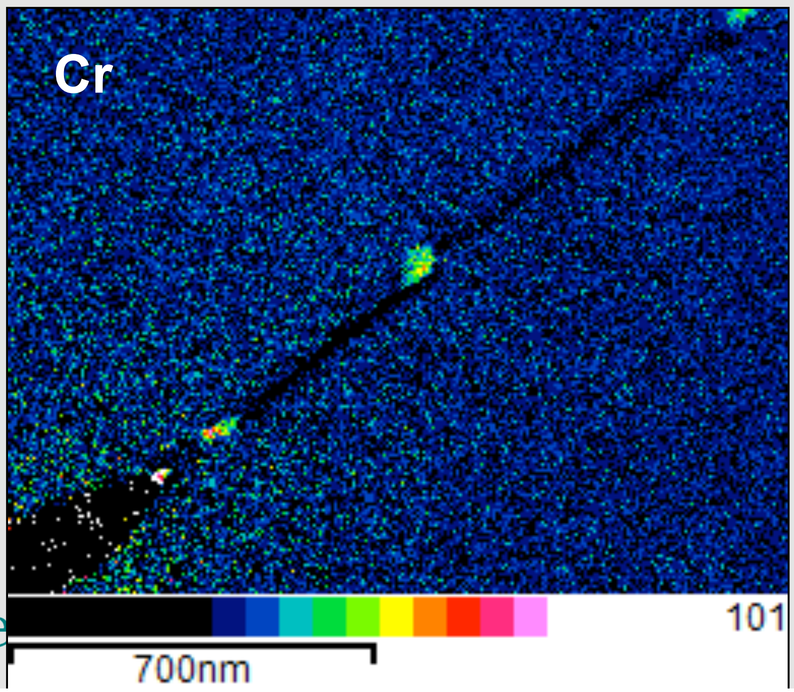


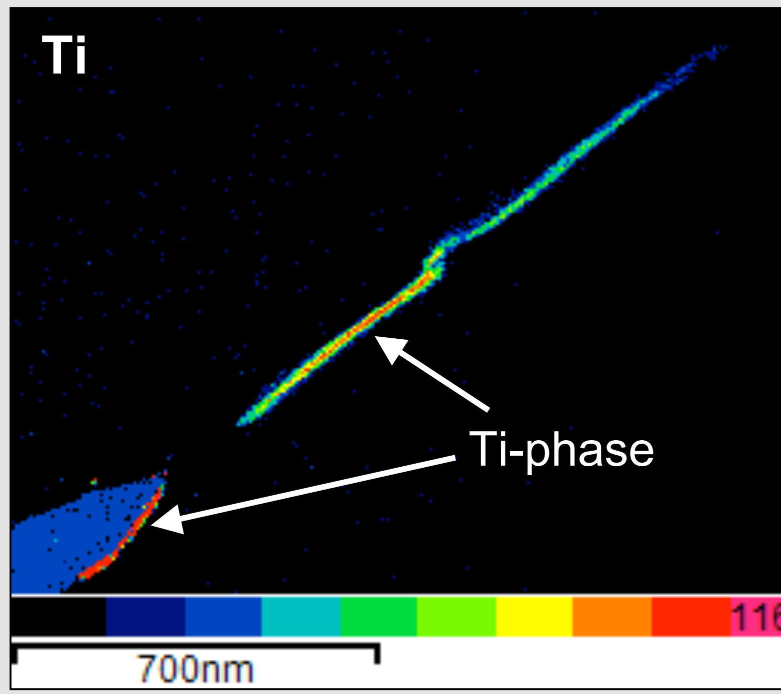




Grain Boundary
TiN phase
MC structure







Alloy 52 Weld Hot Cracks

- *Preliminary examinations document cracking along grain boundaries in the dilution zone. General region has ~25-40 wt% Fe, 35-45 wt% Ni and 23-28% Cr.*
- *Grain boundaries examined in this region have a semi-continuous distribution of thin, elongated TiN nitrides (20-50 nm wide, up to µm in length) and spaced, slightly larger $Cr_{23}C_6$ carbides.*
- *IG crack path appears to be preferentially along TiN interfaces because of distribution, role of $Cr_{23}C_6$ uncertain. Examinations are continuing.*

Major Task Areas

- **Crack-Growth System Construction/Testing**
  - CGR systems and validation testing completed in 2006.
- **Crack-Growth Rate Testing**
  - Austenitic Stainless Steels
  - Alloy 600 and Alloy 182 Weld Metal
  - ***Alloy 690 and Alloy 152/52 Weld Metals***
- **Characterization and Cracking Mechanisms**
  - OM, SEM, EBSD and TEM examinations of test materials
  - SEM/ATEM characterizations of SCC cracks and crack tips in LWR service materials and laboratory CGR samples
  - SEM/ATEM characterizations of hot cracks in mockup and laboratory alloy 182/82 and alloy 152/52 weldments