SCC Crack Growth Rates for Nickel-Base Alloys in PWR Primary Water

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NRC Project N6007

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Pacific Northwest National Laboratory

Presentation Topics

- **Laboratory SCC Crack-Growth Testing**
- **Crack-Growth Rates for Alloy 690/152/52**
  - Current Alloy 690/152/52 Testing and Data Summary
  - Potential Issue for 1D-Rolled Alloy 690
  - Questions, Concerns, Issues and Research Needs
- **Crack-Growth Rates for Alloy 600/182/82**
  - Alloy 600 Data and Disposition Curve
  - Alloy 182/82 Data and Disposition Curves
  - Questions, Concerns, Issues and Research Needs
- **SCC Crack-Growth Testing Issues**
  - System Requirements and Test Approach
  - Problems/Issues for SCC-Resistant Materials

SCC Crack Growth Testing

Crack-growth-rate testing is the most effective and quantitative method to evaluate material and environment effects on SCC. Can be used to:

(1) define and quantify material-environment-stress dependencies on SCC

(2) generate data for mechanistic understanding and form the basis for engineering prediction

(3) resolve confusion and help elucidate service failures

(4) probe new phenomena and help confirm effectiveness of mitigation approaches.

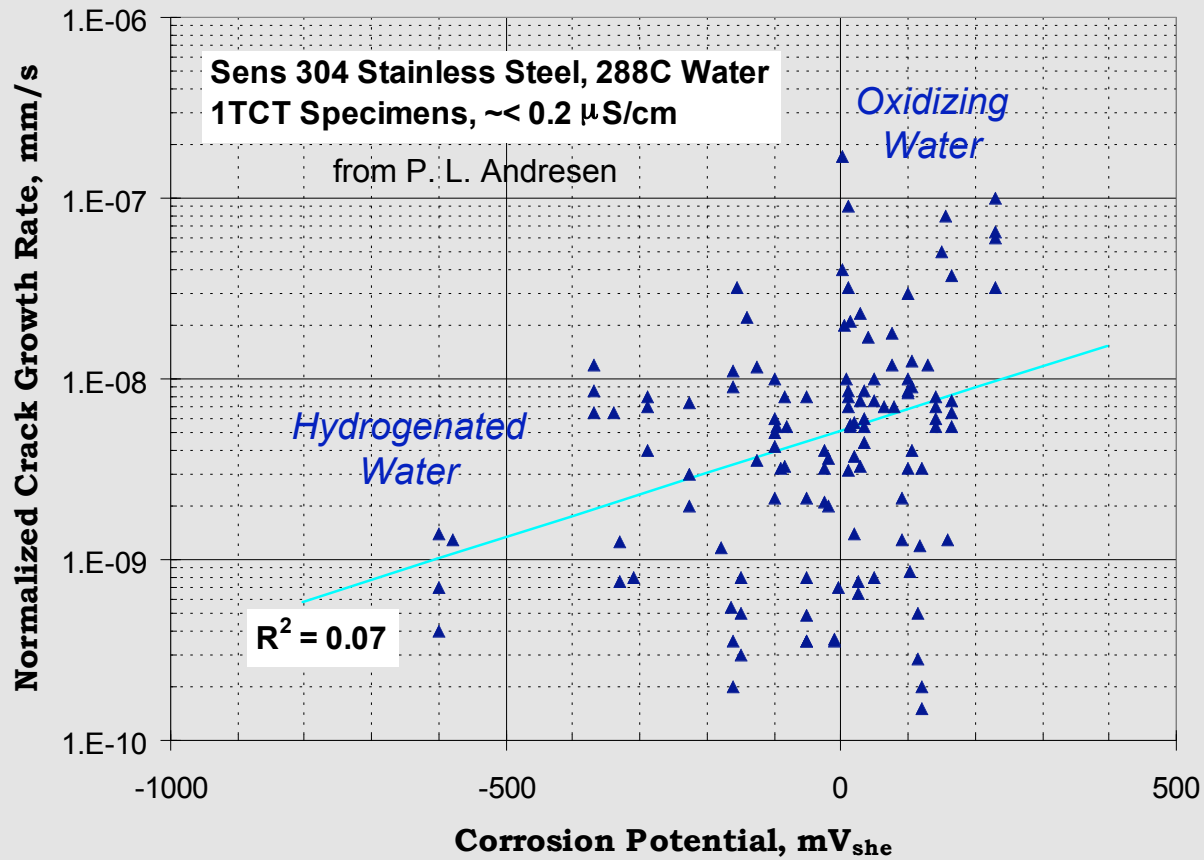
SCC Crack Growth Testing Issues

Many experimental, materials, enviromental and interpretation elements necessary for effective measurement/analysis of SCC:

1. *Successful SCC testing requires a broad knowledge of metallurgy, mechanics, chemistry, electrochemistry, and physical measurements - excellence not in a few areas, but in all*
2. *Test methodology important - transitioning, unloading, K/size, crack front evenness, dcpd resolution, test management*
3. *Material (heat, processing, homogeneity, heat treatment, inclusion/carbides, cold work/HAZ, orientation, …) and environment (temperature, pH, purity, …) variations important*
4. *Interpretation important – uneven crack fronts & data correction, use of avg vs. max CGR, K correction, data reproducibility…*

SCC Testing & Data Issues

*CGR data, even for "well behaved" material, often shows **large scatter** due to testing problems*



Even with best empirical model, scatter can produce a very poor statistical fit. Statistics can't overcome bad experiments.

"quoting" Peter Andresen, GE Global Research

Reproducibility of SCC CGR Data Depends on Many Items Including Test System Capabilities:

1 – Loading stability, dK/da correction, high-to-low frequency cycling, cycle + hold, constant displacement control, …

2 – DCPD crack length measurement resolution & accuracy (need at least <10 μm, <2 μm for SCC resistant alloys)

3 – Temperature (<0.2°C fluctuation), water pressure, seal friction and water chemistry (<0.1 μS/cm outlet) control

4 – Reference electrode accuracy & reliability

5 – Ability to make changes "on-the-fly", e.g., in temperature, H_2 concentration, pH/B/Li/impurities, …

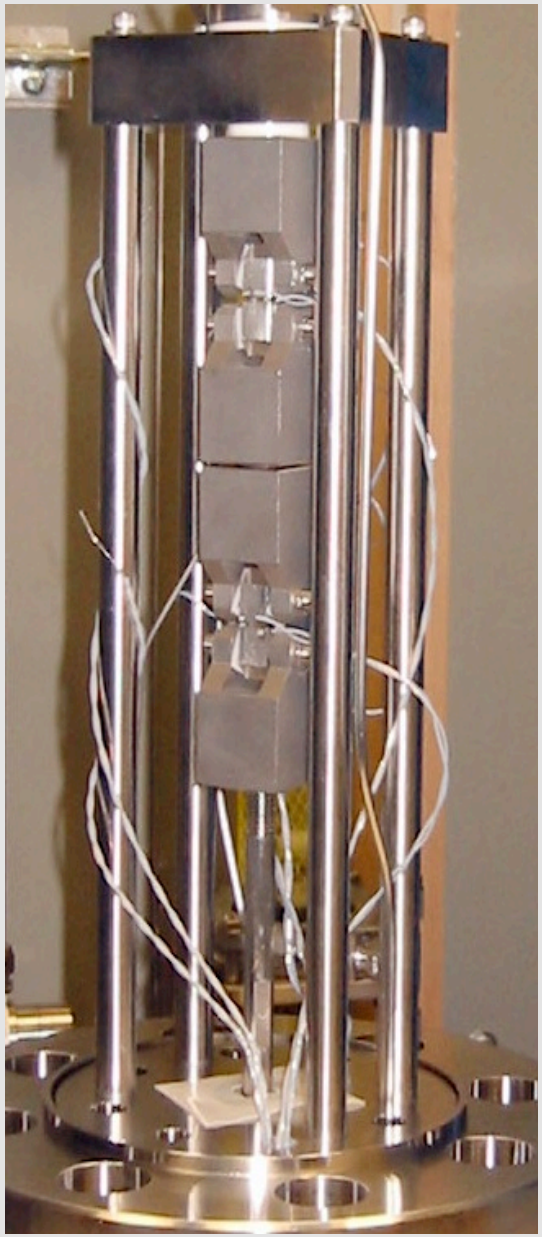
6 – Maintain continuous operation and stability of all test conditions over long times (>12 months)

PNNL SCC Crack Growth Test Systems

- *Outlet conductivity ≤0.065 µS/cm under BWR test conditions*
- *Reversing DCPD, automated K control, autoclave flow rate ensures that outlet water chemistry close to inlet chemistry*
- *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

- *PWR Primary Water Conditions: 1000 ppm B, 2 ppm Li, 11-29 cc/kg H_2 and test temperature 280-350°C.*
- *Single sample fatigue precracked in situ, 2-3 samples in series precracked ex situ; typical K = 10-40 MPa√ m*
- *Crack transitioning in situ: decreasing fatigue cycle frequency; then 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.*



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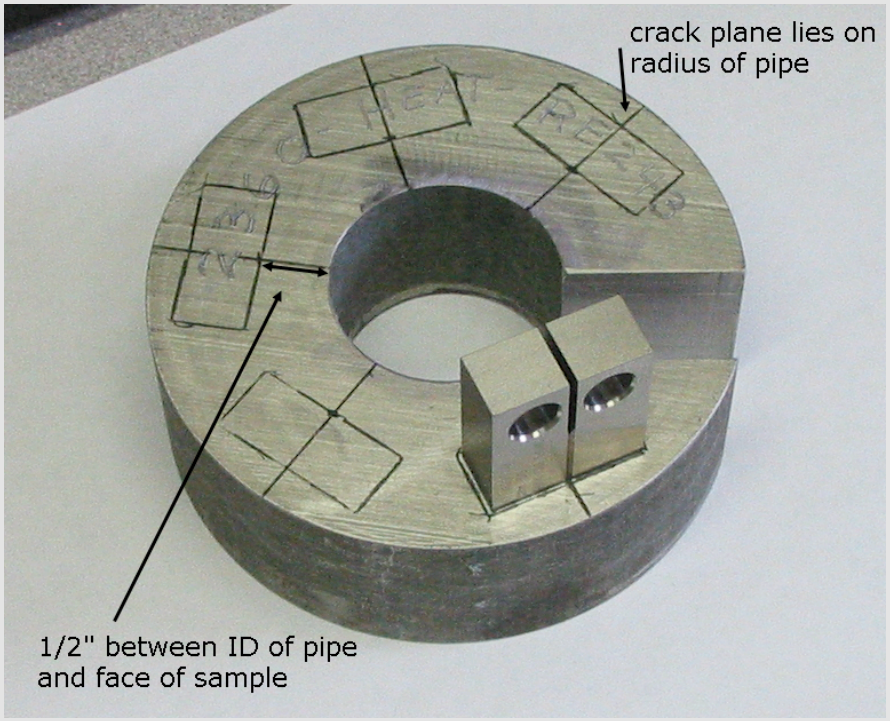
Alloy 690/152/52 Sources of Data

- *PNNL - actively loaded CT*
- *GE Research - actively loaded CT*
- *ANL - actively loaded CT*
- *Bettis - primarily bolt loaded CT*
- *INSS - constant load CT*
- *EdF - RUB, DCB, CT*

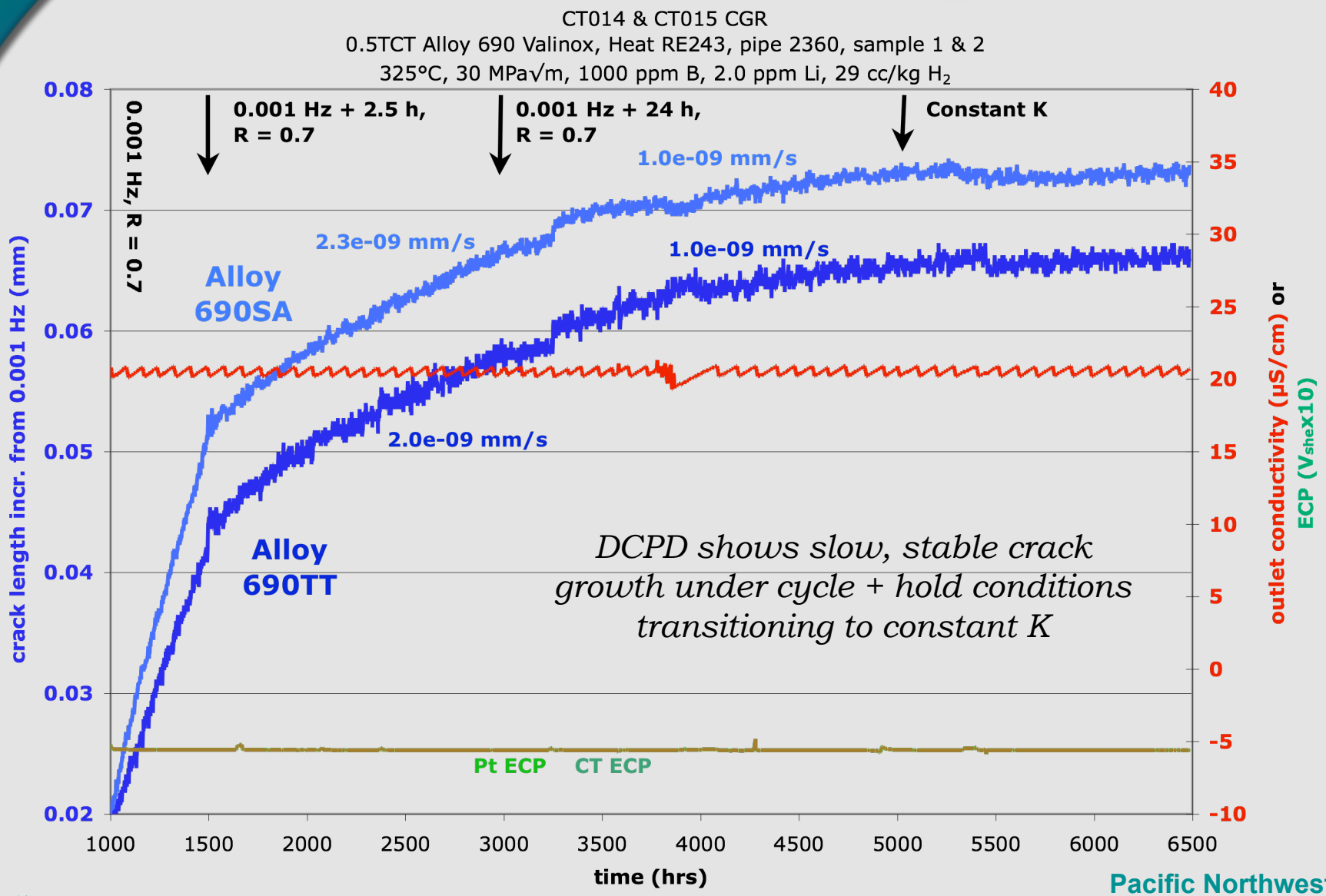
Alloy 690 CRDM Materials

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) Crack Transitioning



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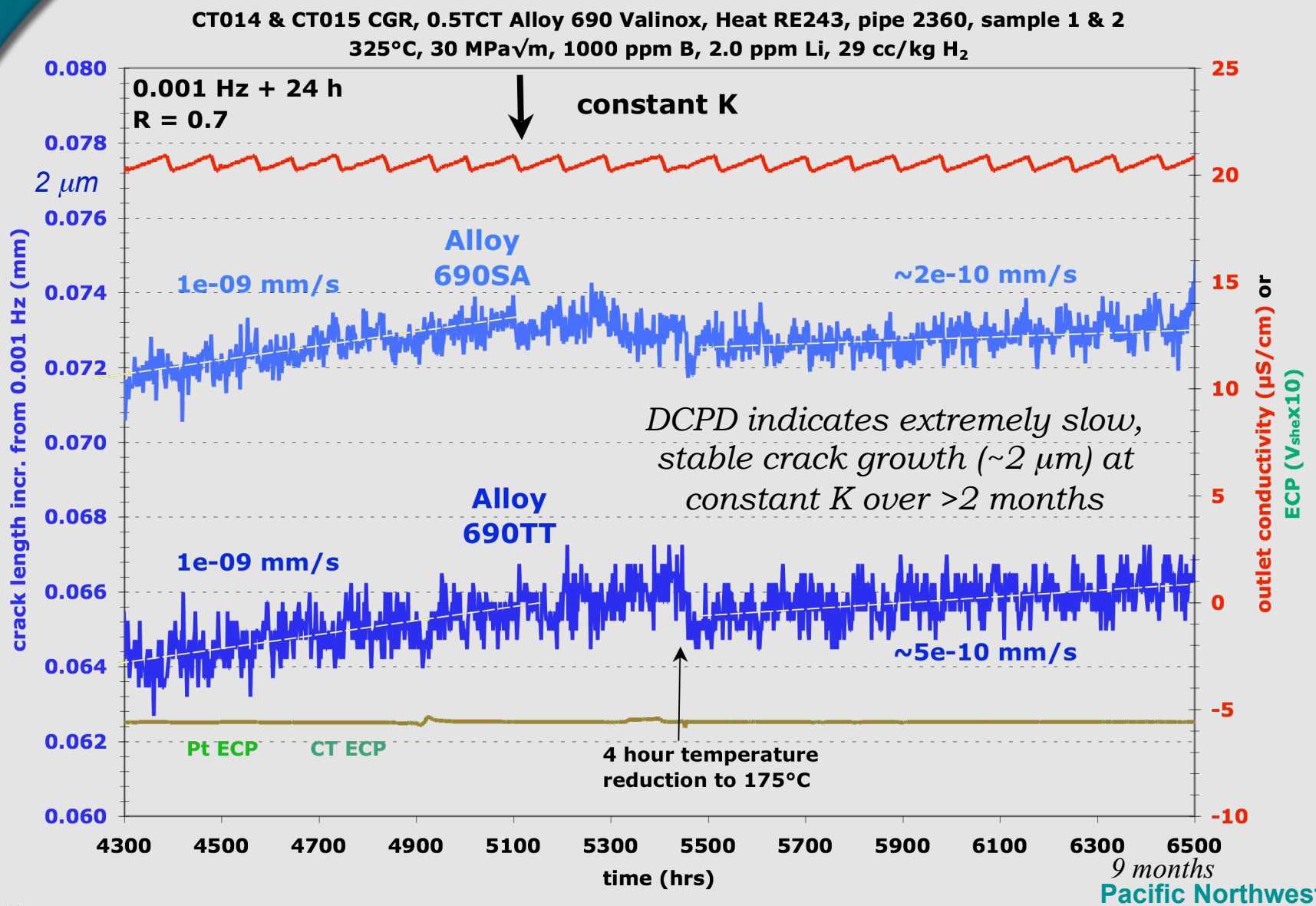
Alloy 690 (TT/SA): Constant K



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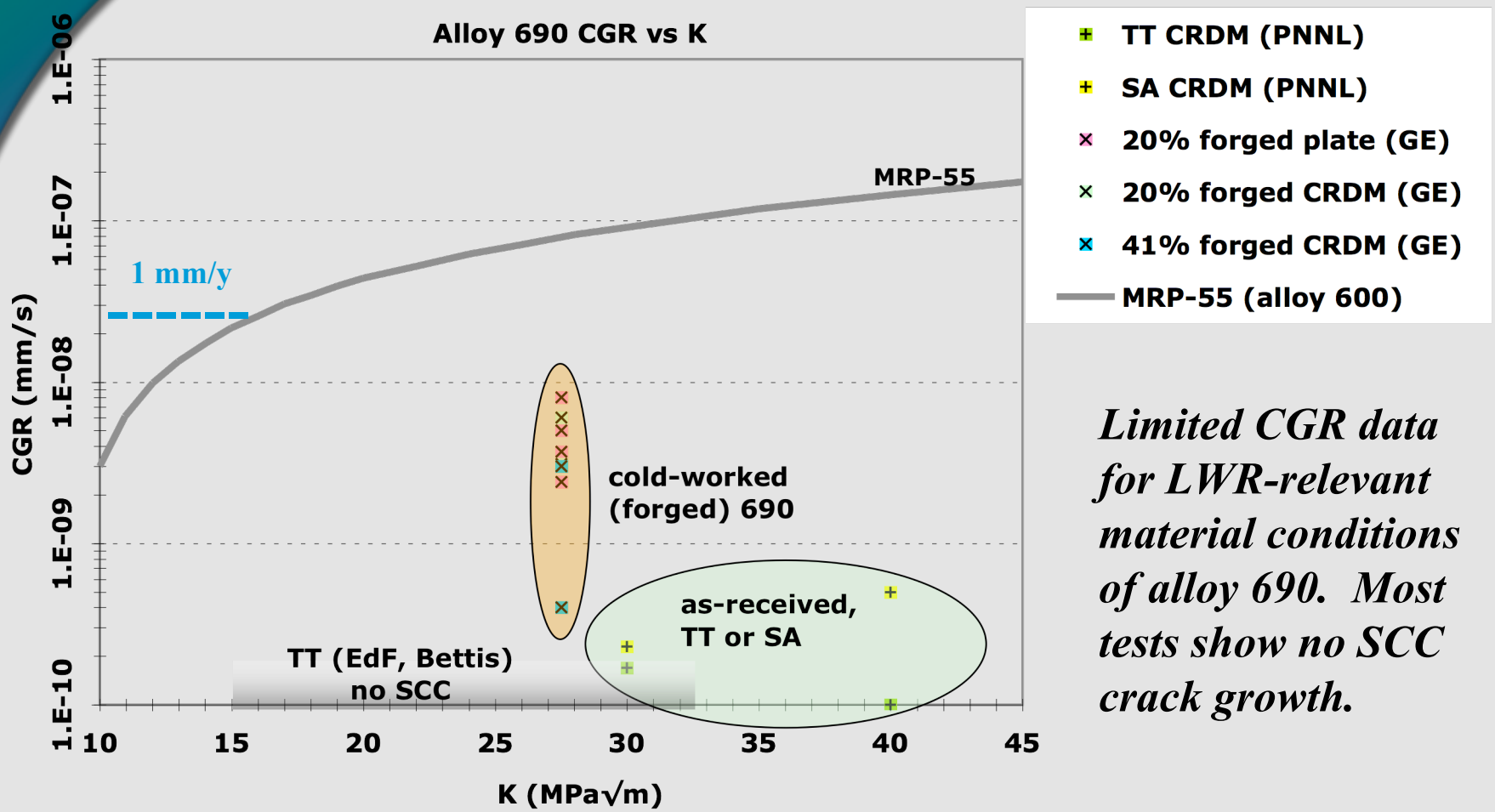
Alloy 690 Summary - PNNL

- *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 SCC crack advance under gentle cycle + hold and constant K conditions, but at extremely low propagation rates. DCPD-measured CGRs under constant K were* $<5x10^{-10}$ *mm/s. SCC response similar at K levels from 30-40 MPa* $\sqrt{m}$.

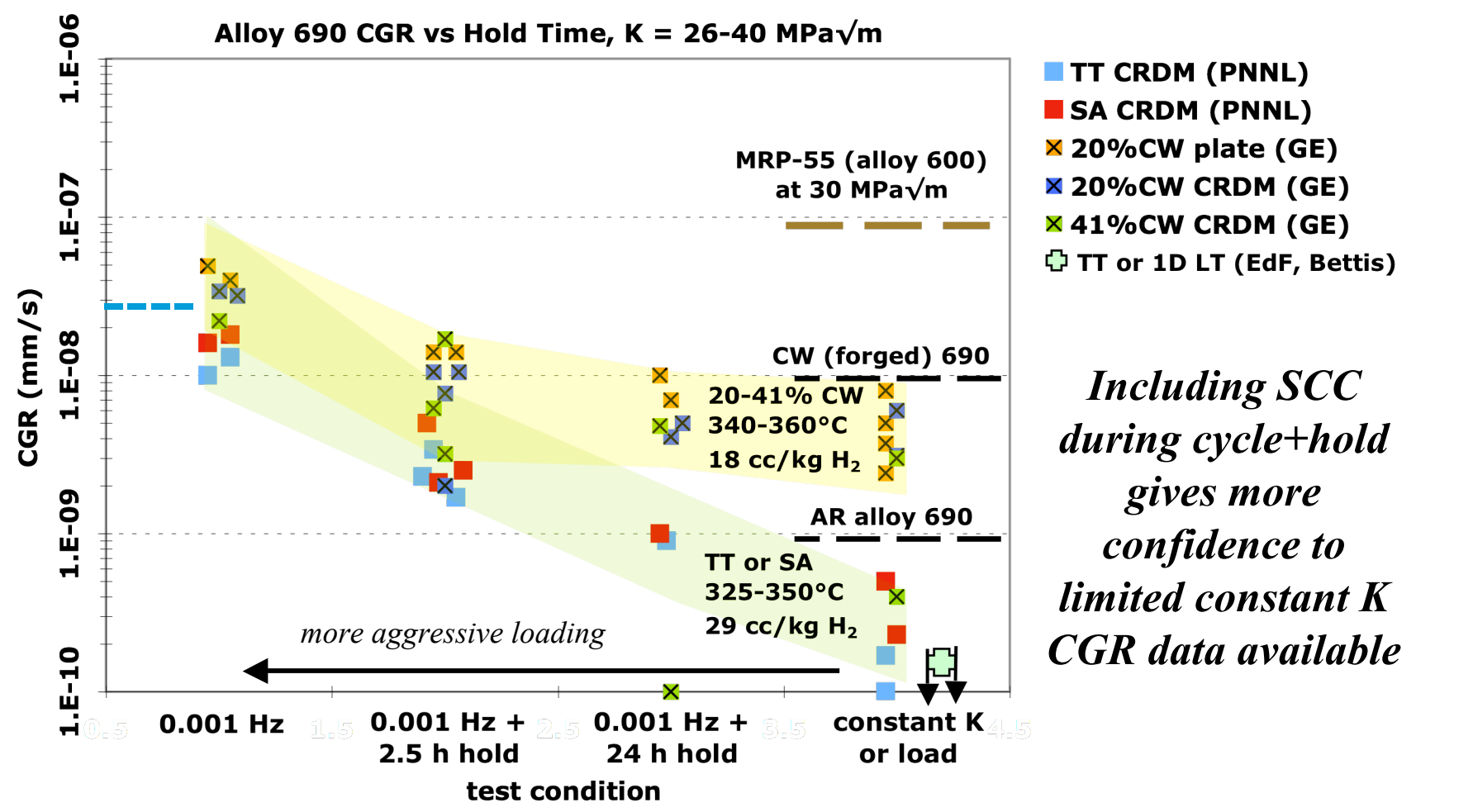
Alloy 690 - K Effects



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

- *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.*
- *Much more high-quality CGR data on alloy 690 required.*

Alloy 690 + Cycle/Hold



Including SCC during cycle+hold gives more confidence to limited constant K CGR data available

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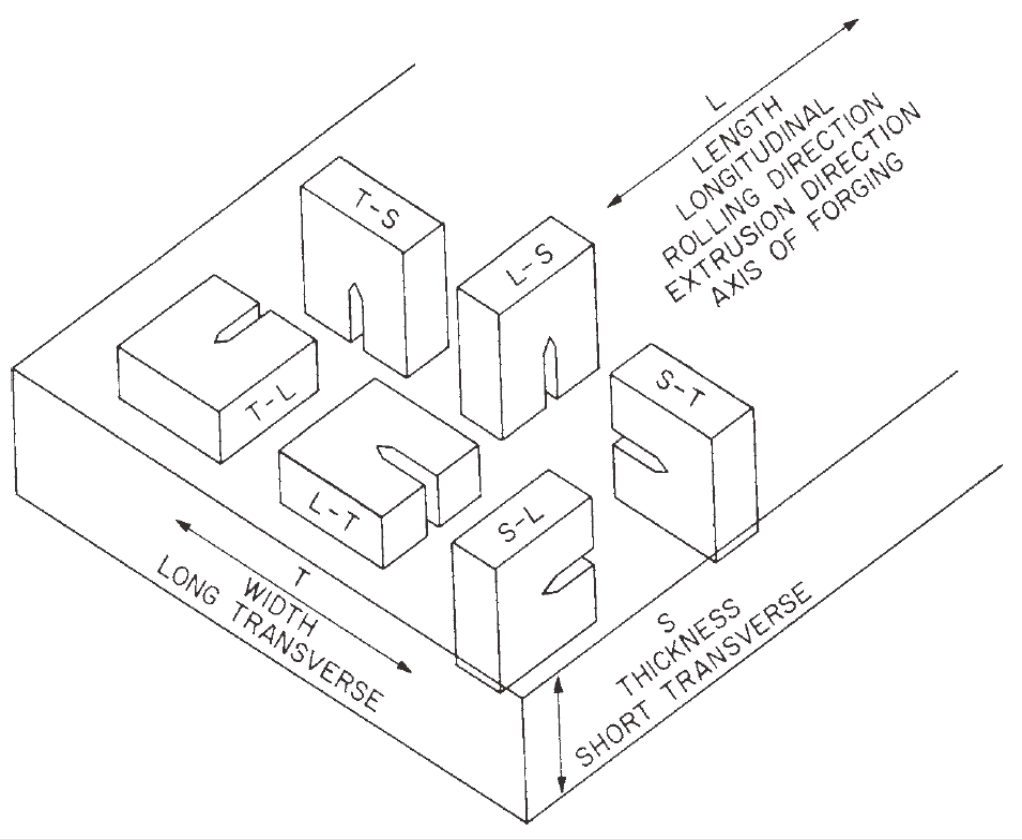
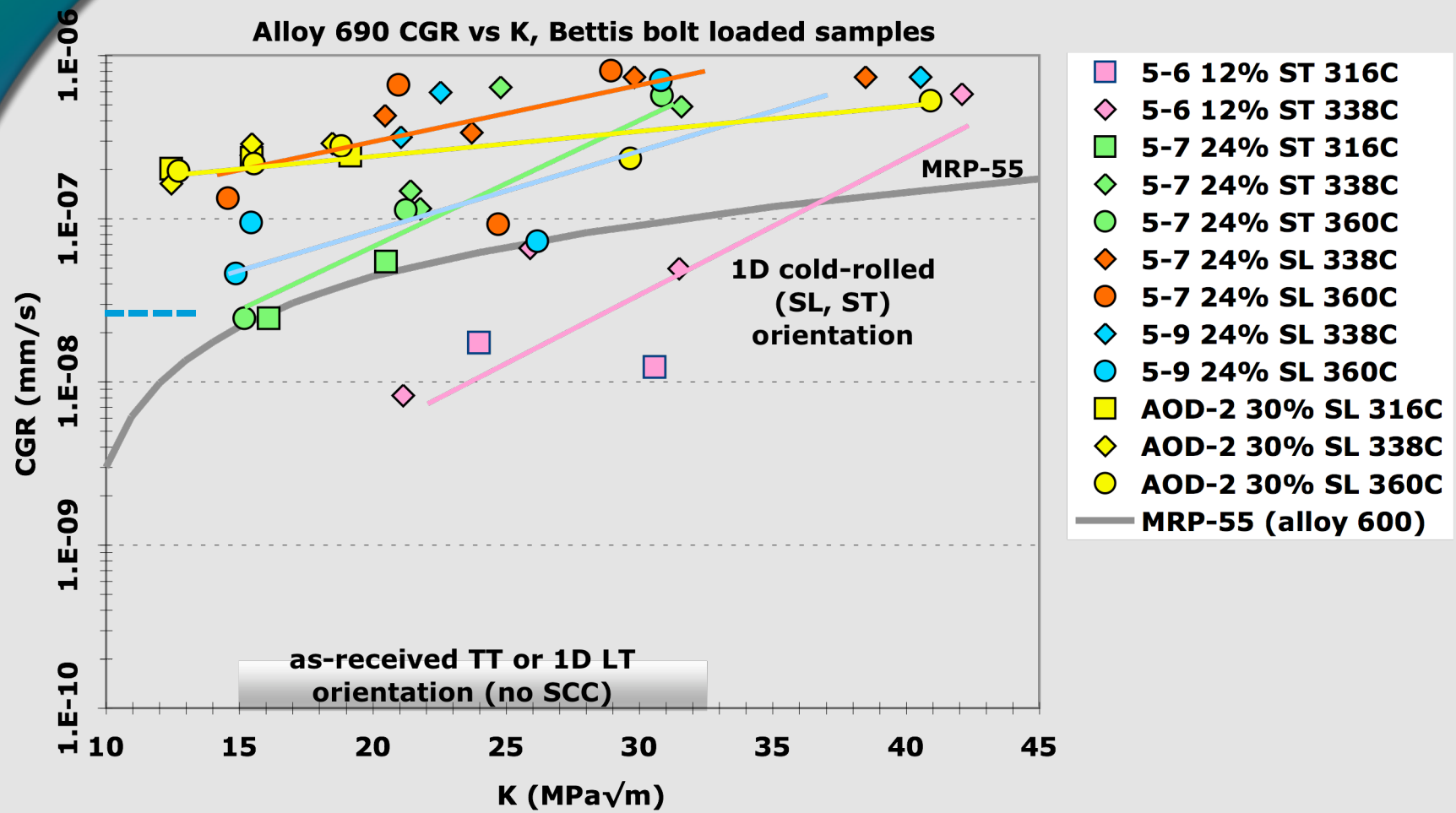
Laboratory CT Specimen Orientations



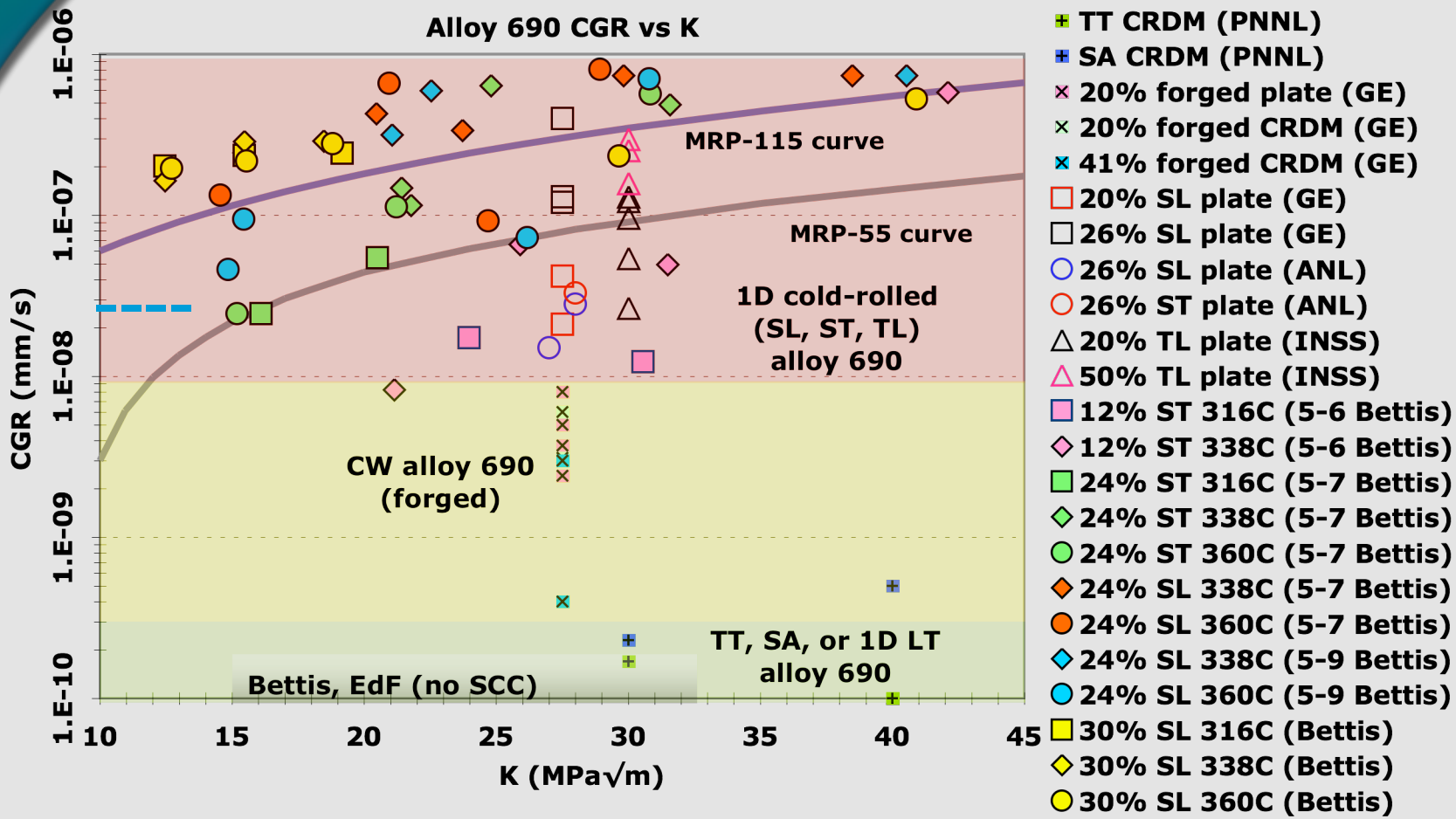
- *Recent results by Bettis, GE Global Research, ANL and INSS have shown very high CGRs for certain specimen orientations when taken from 1D cold-rolled materials.*
- *S-L, S-T and T-L have all shown higher crack growth rates.*

Alloy 690 - Bettis 1D Rolled



- *1D cold-rolled (S-L, S-T, T-L) materials exhibit high CGRs at constant K or constant load even at lower K values.*

All Alloy 690 - 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?*

Increased IGSCC Susceptibility of 1D Cold Rolled Materials

1D cold rolling produces grain elongation, damage and high dislocation densities near grain boundaries, and damage-cracking of second-phase particles.

Inhomogeneity of thru-thickness CW vs. %RA & %RA per pass

Forging and cross-rolling are probably not immune from these problems, but appear to be less susceptible.

Weld shrinkage strains in the HAZ can be viewed as conceptually as "tensile forging".

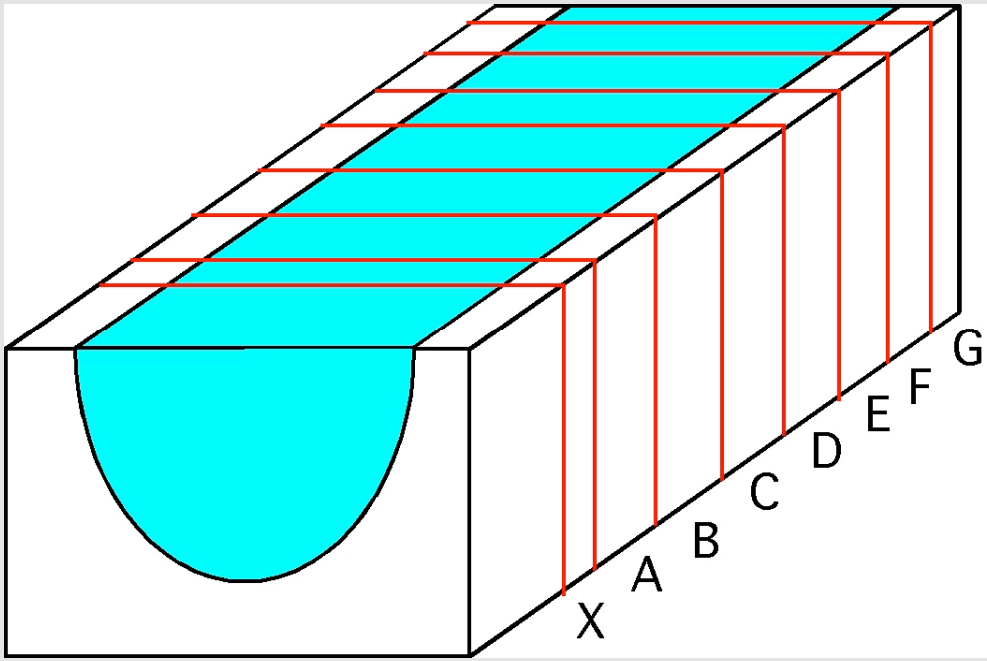
A more complete characterization and understanding of damage evolution from various types of deformation is needed to directly link to increased IGSCC susceptibility.

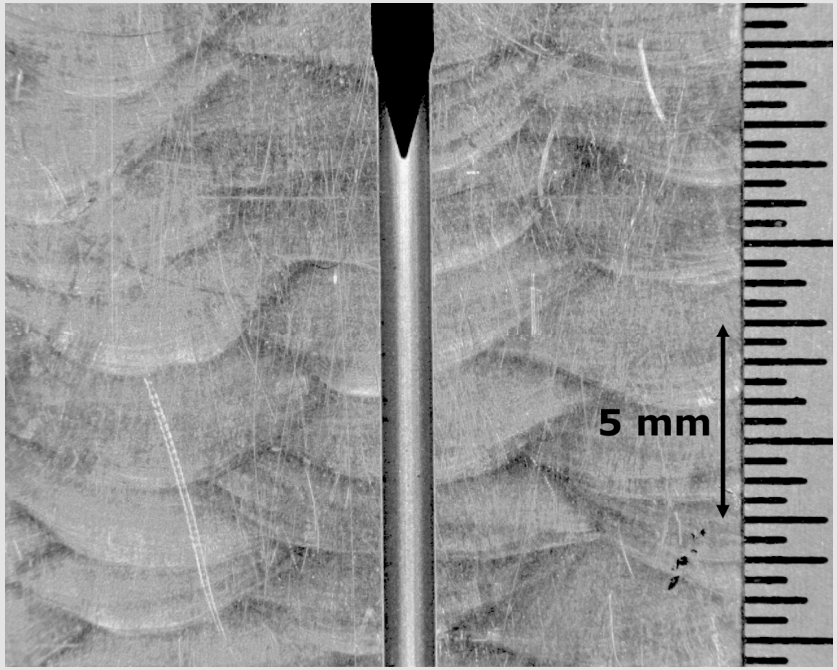
Key Issues for Alloy 690 SCC Crack Growth Response

- **Heat-to-Heat Variability**
  - *Limited number of alloy 690 heats have been evaluated, range of heats and microstructures should be examined*
  - *Some indication that melting practice may influence SCC*
  - *Inhomogeneity & compositional banding may be important*
- **Thermechanical Processing Effects**
  - *Grain boundary segregation and precipitation effects on SCC*
  - *Cold/warm working effects on grain boundaries, IG/TG particles and SCC*
  - *Weld HAZ effects on microstructure, strength and SCC*
- **Relevance of 1D Cold Rolled Material**
  - *What process promotes such high SCC susceptibility?*
  - *How does 1D rolling deformation correlate to 2D deformation during weld shrinkage in alloy 690 HAZ? Will similar SCC susceptibility occur at higher strains in 2D?*

Alloy 152 Mock-Up Weld

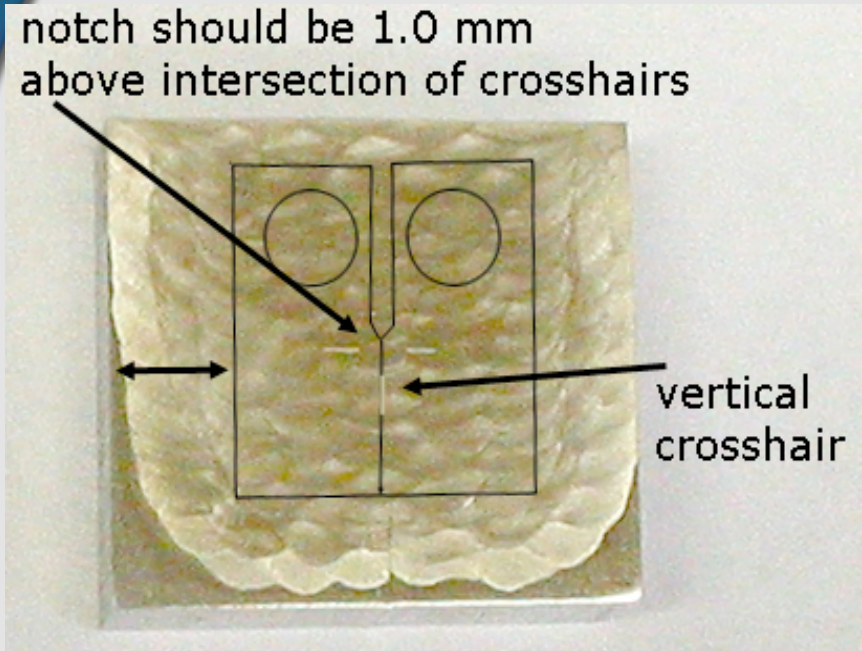
Made using 6.1-cm-thick, 304SS plates, Alloy 152 butter passes and weld obtained from EPRI NDE Center produced by MHI for Kewaunee pressure vessel head replacement

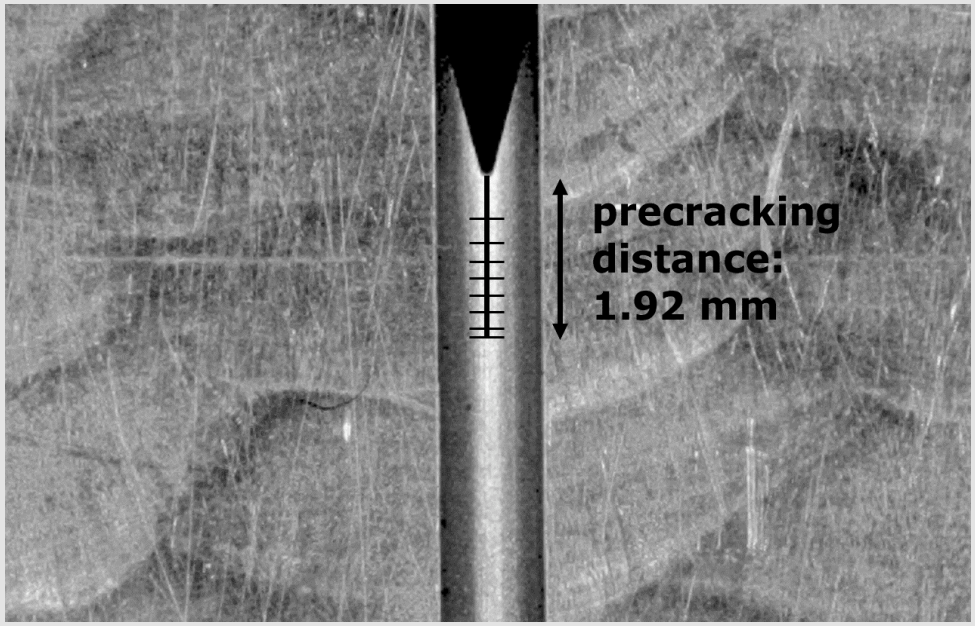




Weldment was sectioned into 1.9-cm thick blocks and surfaces of CT blanks (C, D and E) characterized so that pre-cracks are located in a single weld pass, aligned with elongated dendrites and enable >1 mm of SCC crack growth within the weld pass.

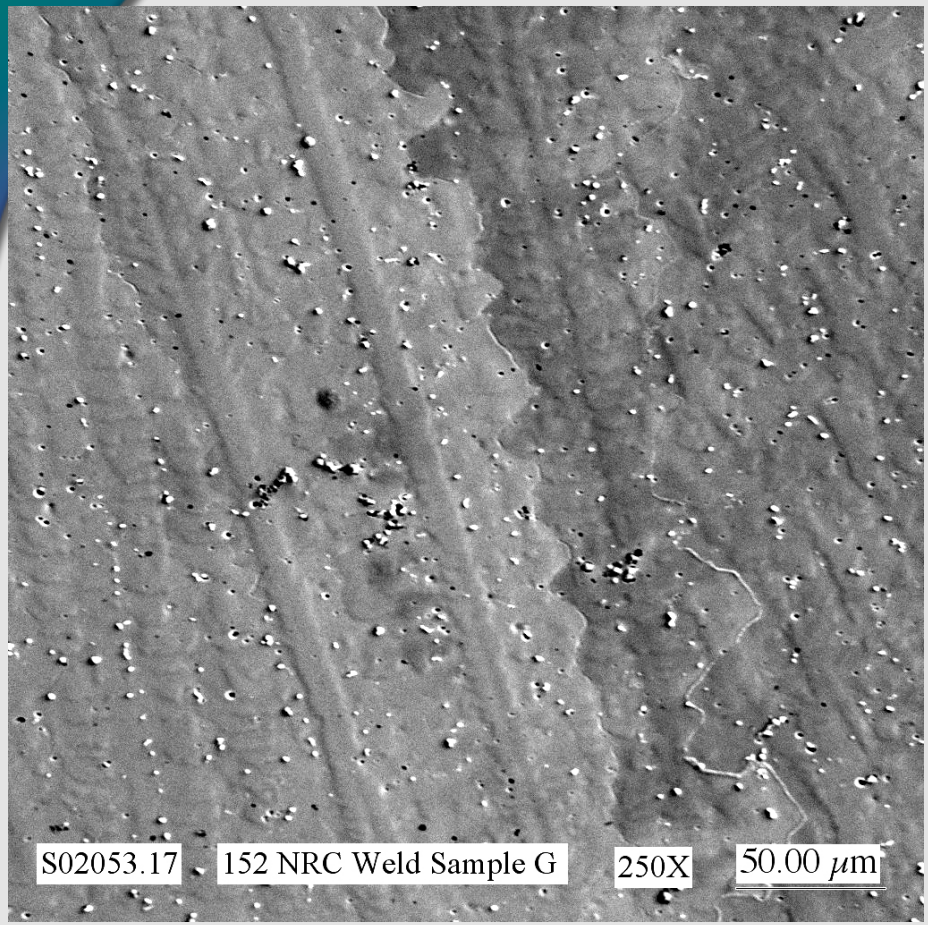
Alloy 152 Weld Metal Specimen

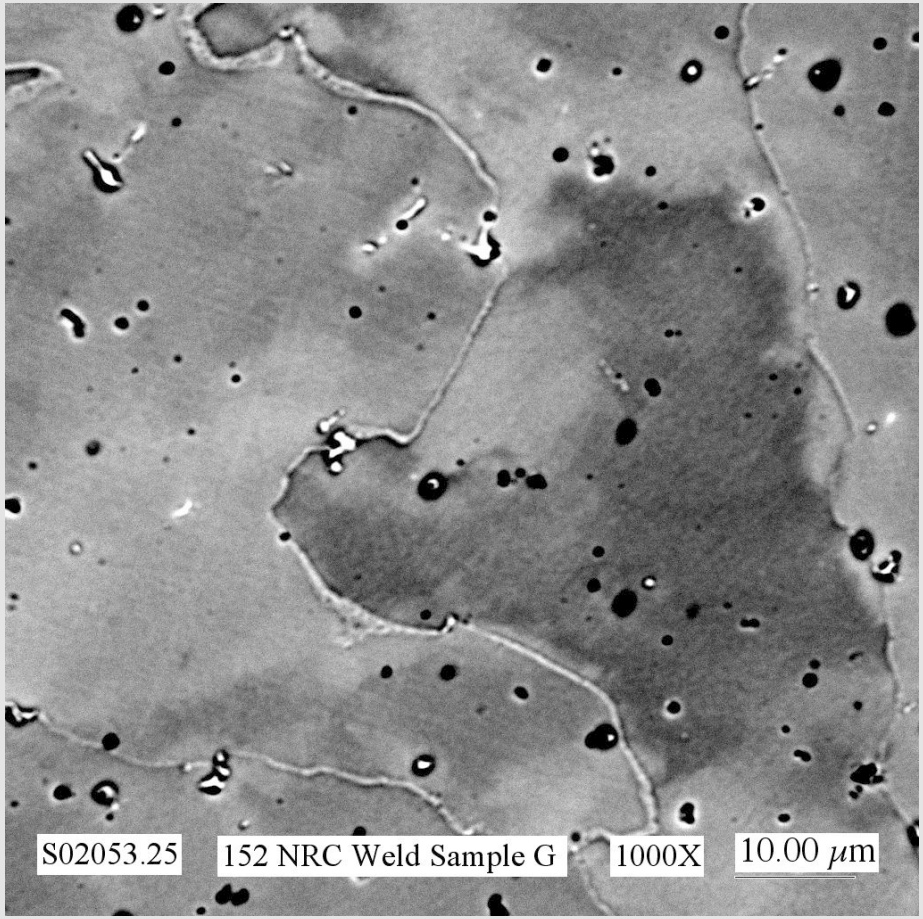




Sample machined entirely of weld metal. Notch and pre-crack oriented to allow SCC growth in the middle of a weld pass with crack oriented roughly along dendrite direction.

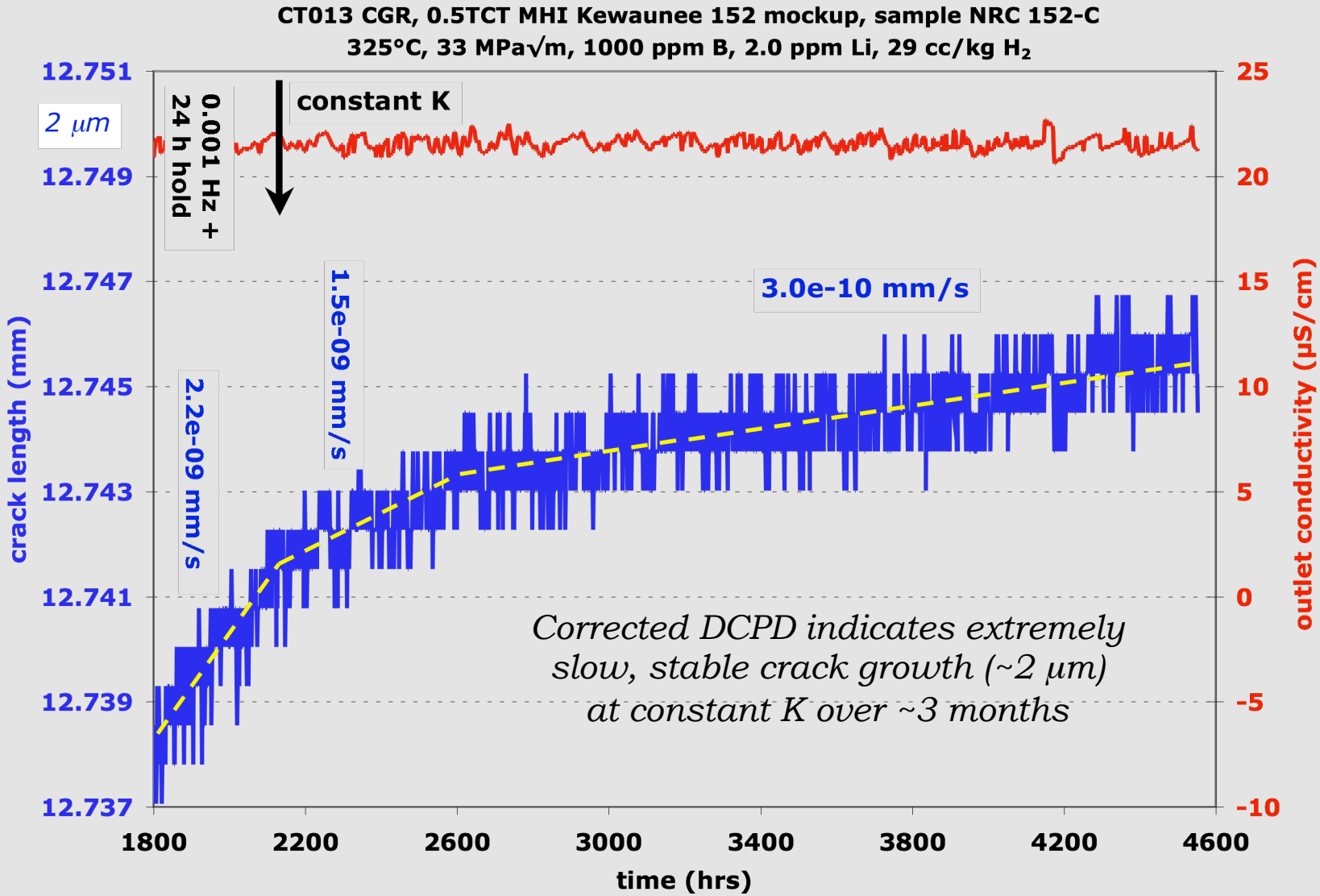
Alloy 152 Weld Metal Microstructure



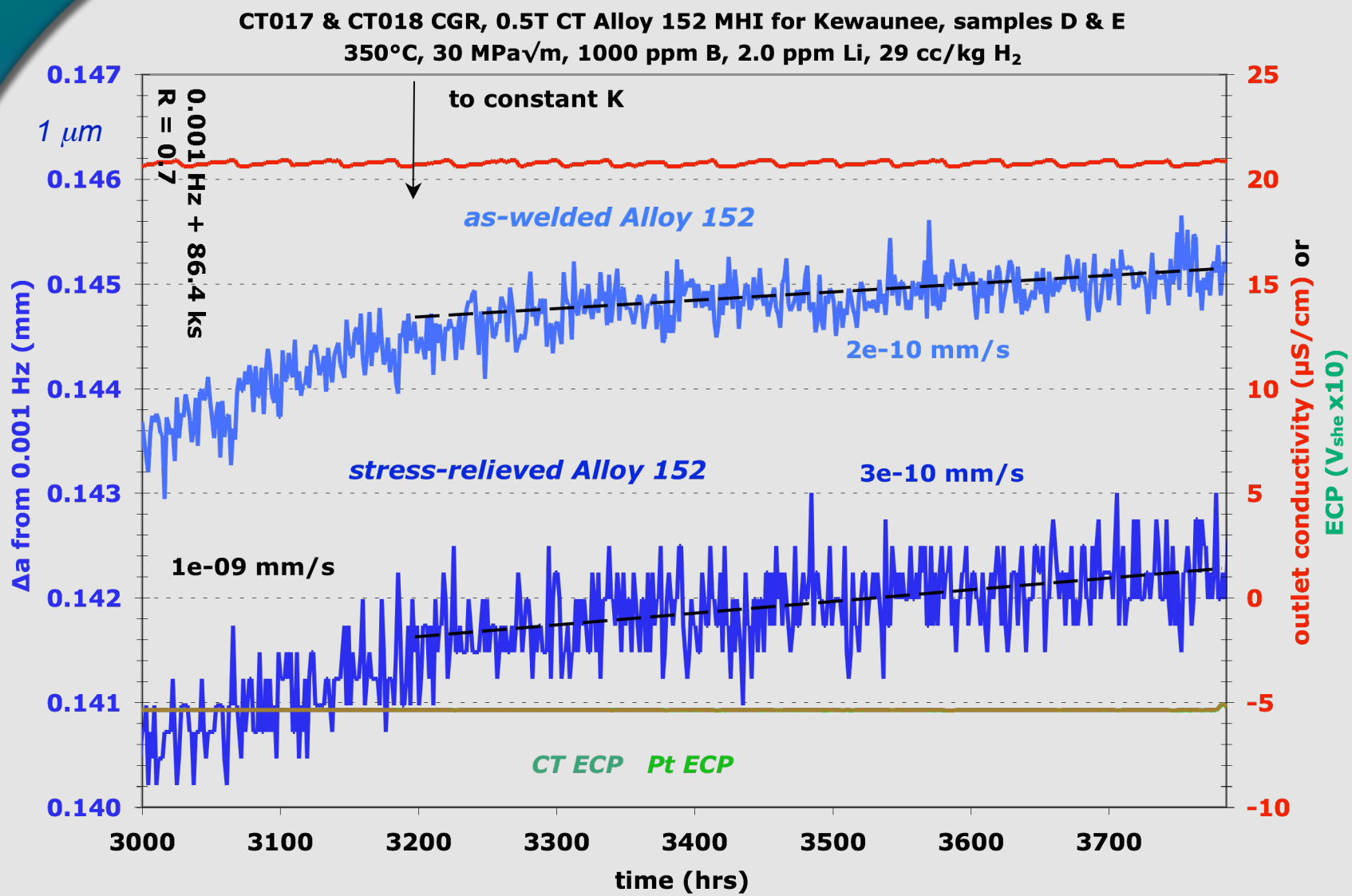


SEM backscatter electron images showing convoluted dendritic boundaries with a high density of Al-Ti-oxide (dark) and Nb-carbide (light) particles throughout the matrix. Fine grain boundary carbides on many grain boundaries.

Alloy 152: Constant K

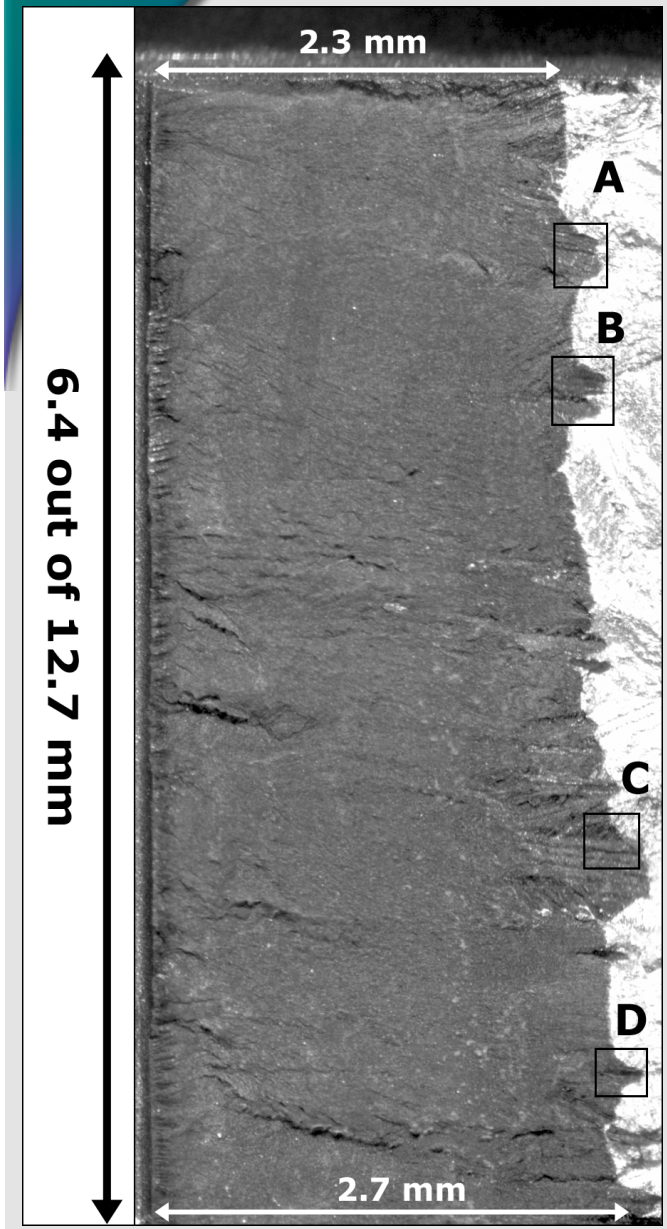


Alloy 152: Constant K

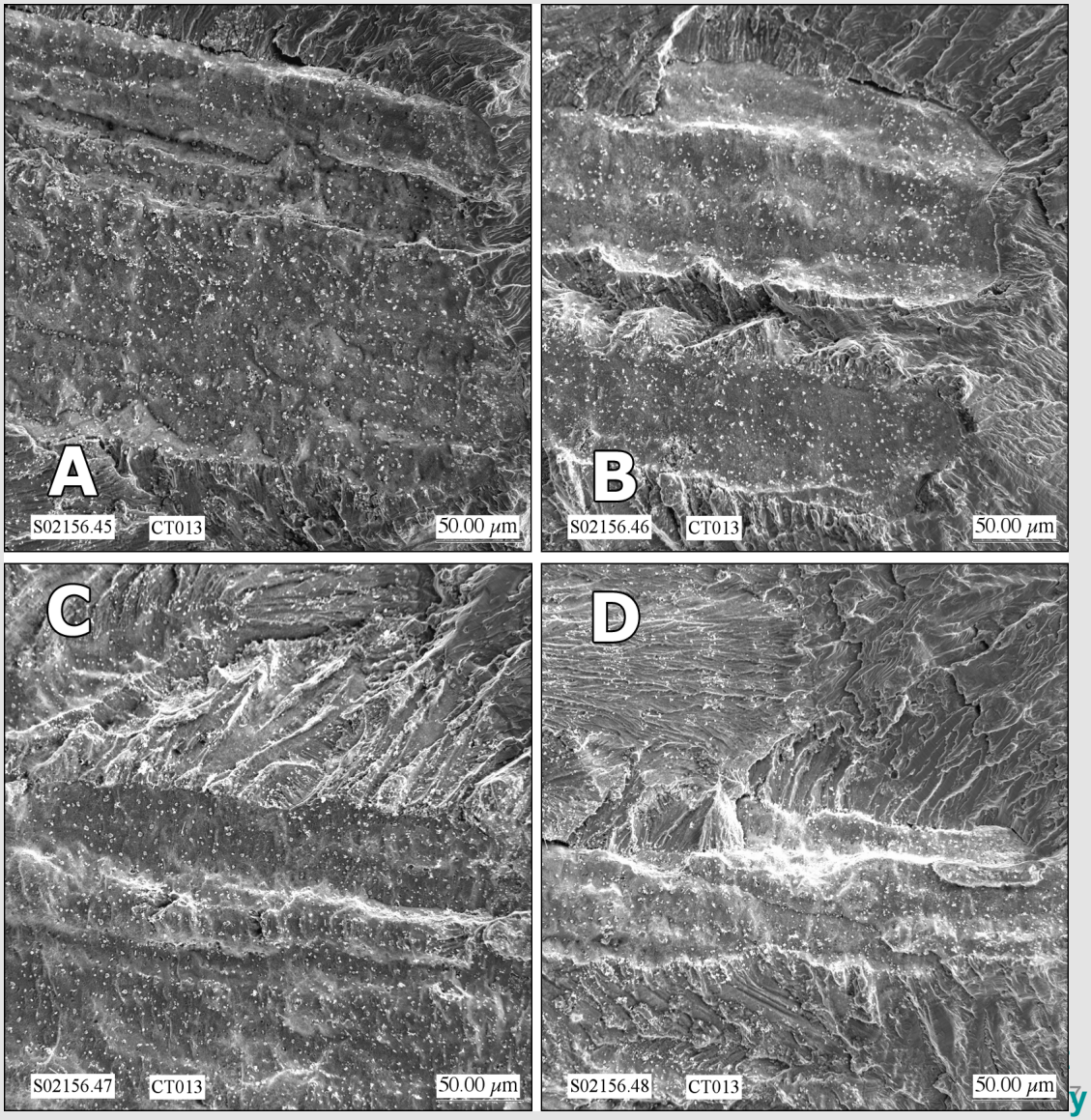


Corrected DCPD indicates extremely slow, stable crack growth (~1 μm) at constant K over ~2 months

Post-Test Fracture Surface



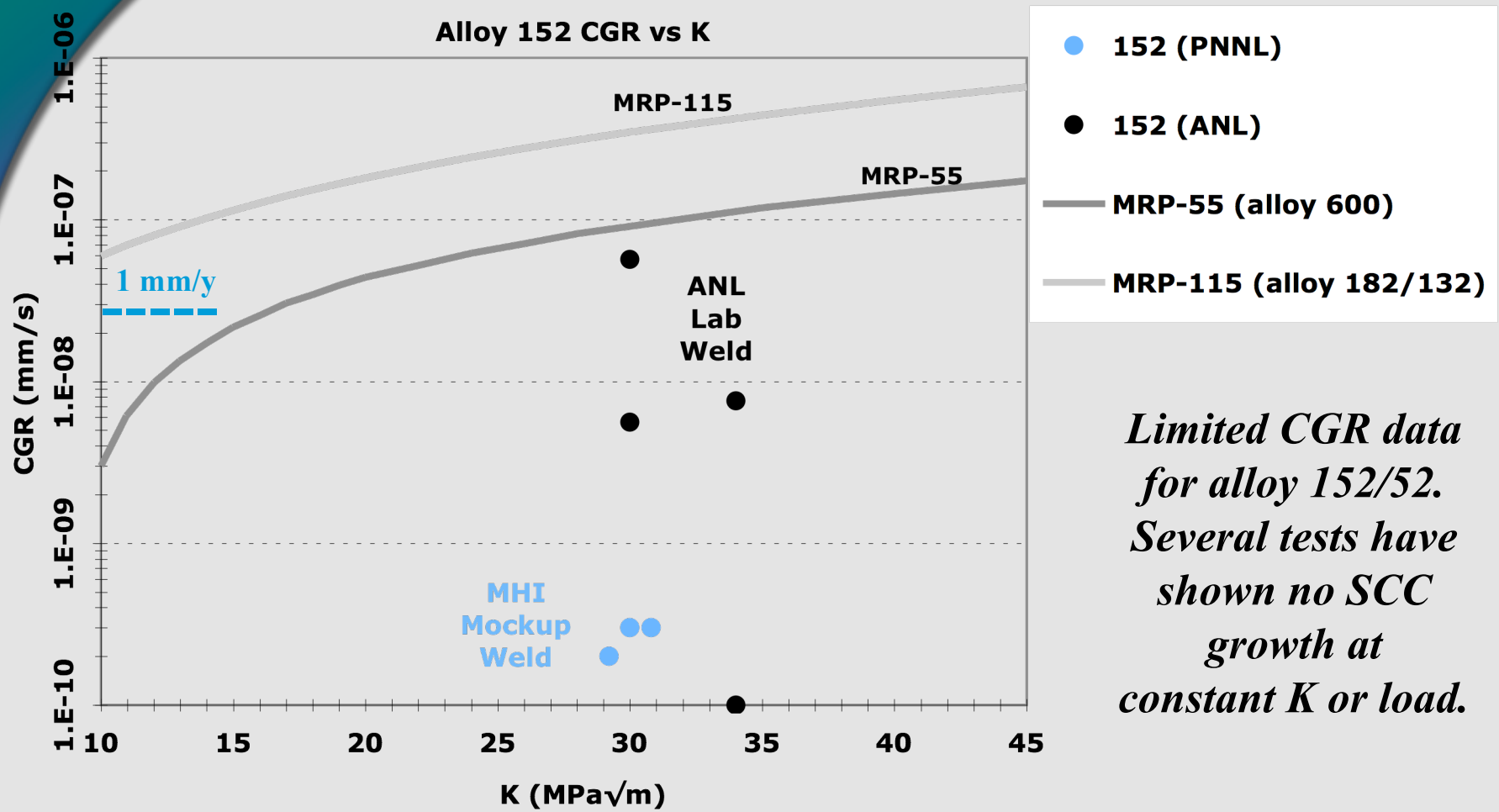
Intergranular SCC seen at crack front



Alloy 152 Summary - PNNL

- *Initial 3 tests show Alloy 152 weld metal resistant to SCC crack growth in simulated primary PWR water at 325 or 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 extremely low propagation rates.*
- *Fractography reveals IGSCC during the cycle + hold and constant K loading conditions. Adjusted average CGRs under constant K ranged from 2-5x10^{-10} mm/s.*

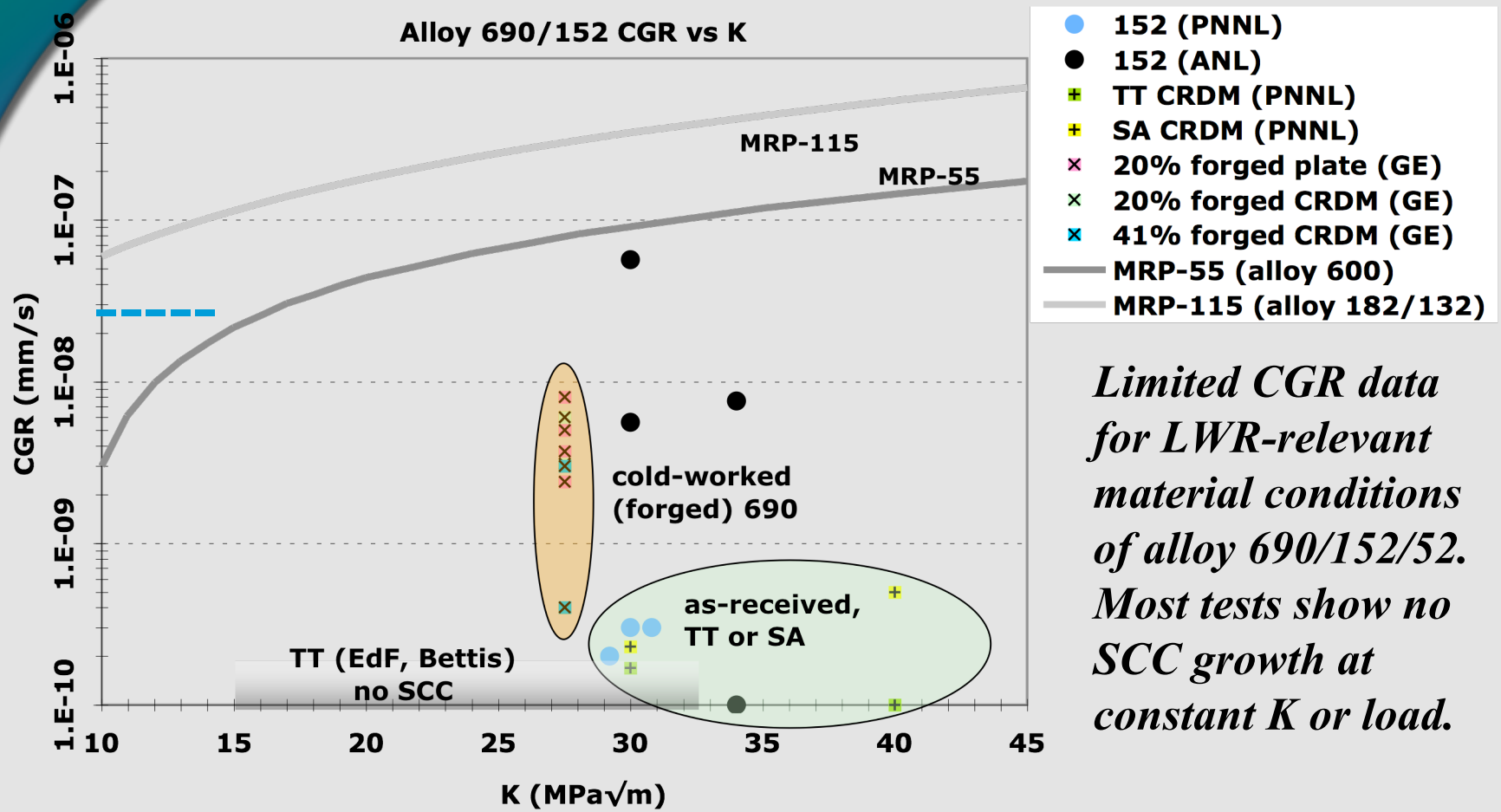
Alloy 152/52 - Limited CGR Data



Limited CGR data for alloy 152/52. Several tests have shown no SCC growth at constant K or load.

- *Much higher CGRs have been reported by ANL on an Alloy 152 laboratory weld.*
- *Obviously much more CGR data required for high Cr, Ni-base weld metals, e.g., alloy 152/52/52M.*

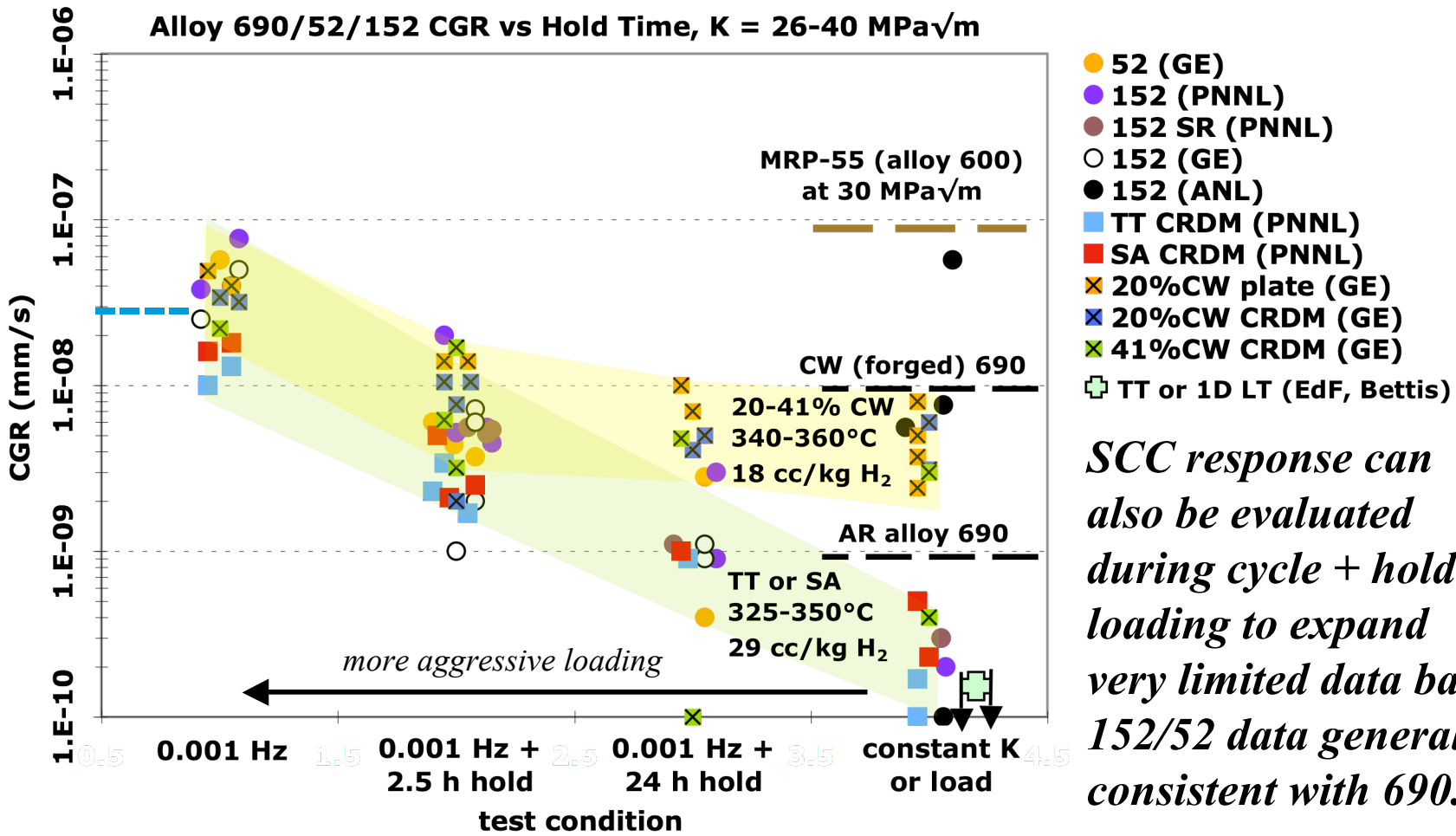
Alloy 690/152/52



Limited CGR data for LWR-relevant material conditions of alloy 690/152/52. Most tests show no SCC growth at constant K or load.

- *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.*
- *Much more high-quality CGR data on alloy 690/152/52 required.*

Alloy 690/152/52 + Cycle/Hold



SCC response can also be evaluated during cycle + hold loading to expand very limited data base. 152/52 data generally consistent with 690.

- *CGRs still low even with low Hz (gentle) cycling + hold.*
- *Worked alloy 690 have higher CGRs across range of conditions.*
- *Upper bound CGRs: ~10^{-9} mm/s as received, ~10^{-8} mm/s CW.*

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Key Issues for Alloy 152/52 SCC Crack Growth Response

- **Weld-to-Weld/Overlay/Inlay Variability**
  - *Very few alloy 152/52 weld metals have been evaluated, greater range of welds and microstructures should be examined*
  - *Different weld types and welding techniques will alter weld shrinkage strains, microstructural evolution, …. (SCC?)*
- **Dissimilar Welds - Dilution Zones**
  - *Critical Cr concentration needed for improved SCC resistance, addition evaluation needed for alloy 152/52 welds with lower Cr, Ni-base alloys (600/182/82).*
  - *More complex composition changes occur in dilution zones between alloy 152/52 and stainless steels. How will Fe and minor elements/impurities from SS influence SCC?*
- **Weld Hot Cracks**
  - *Will presence of small hot cracks in main weld or in dilution zones influence SCC propagation?*
  - *Potential for altered chemistry in solidification hot cracks, may alter crack tip environment and SCC.*

PNNL FY08 Research Plans: Alloy 690/152/52

➢ Alloy 152/52 Weldments

- Three tests on MHI alloy 152 mockup weld completed and tests starting on alloy 52 MHI and AREVA alloy 52 mockups.
- Begin tests evaluating regions in the alloy 152 butter passes and near alloy 152 - SS fusion zone within the dilution zone.
- Hot cracks being characterized in alloy 52 weld and in 52/SS dilution zone plus SCC crack tips in alloy 152.
- Obtaining alloy 52 overlay and inlay mockups from Ringhals for characterization and possible SCC testing.

➢ Alloy 690 CRDM Tubing Heats

- Continue tests on alloy 690 CRDM heats for as-received TT and SA conditions in 1D cold rolled (CR) materials (17-30%).
- Tests starting on 3 additional alloy 690 CRDM heats all in the as-received TT condition.
- Alloy 690 HAZ being characterized in the alloy 52 weld for possible CGR test samples plus SCC cracks tips in alloy 690 (as received vs 1D cold rolled).

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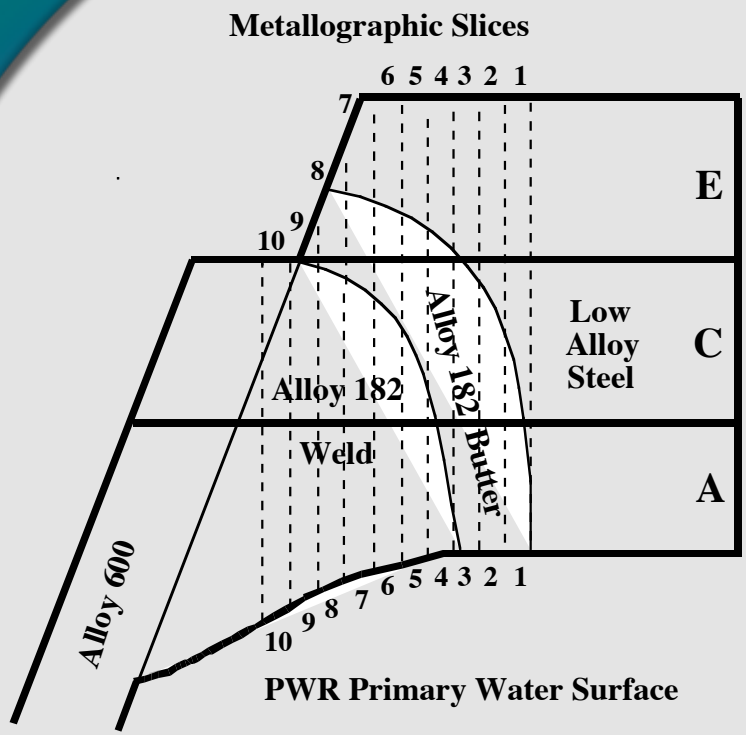
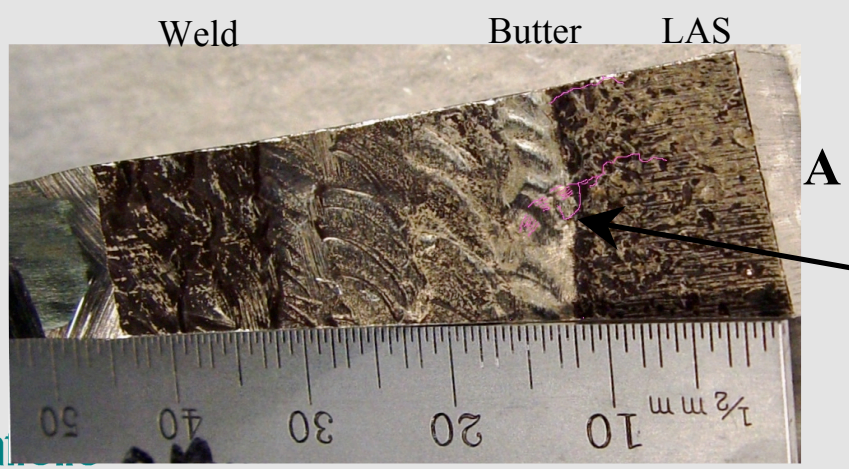
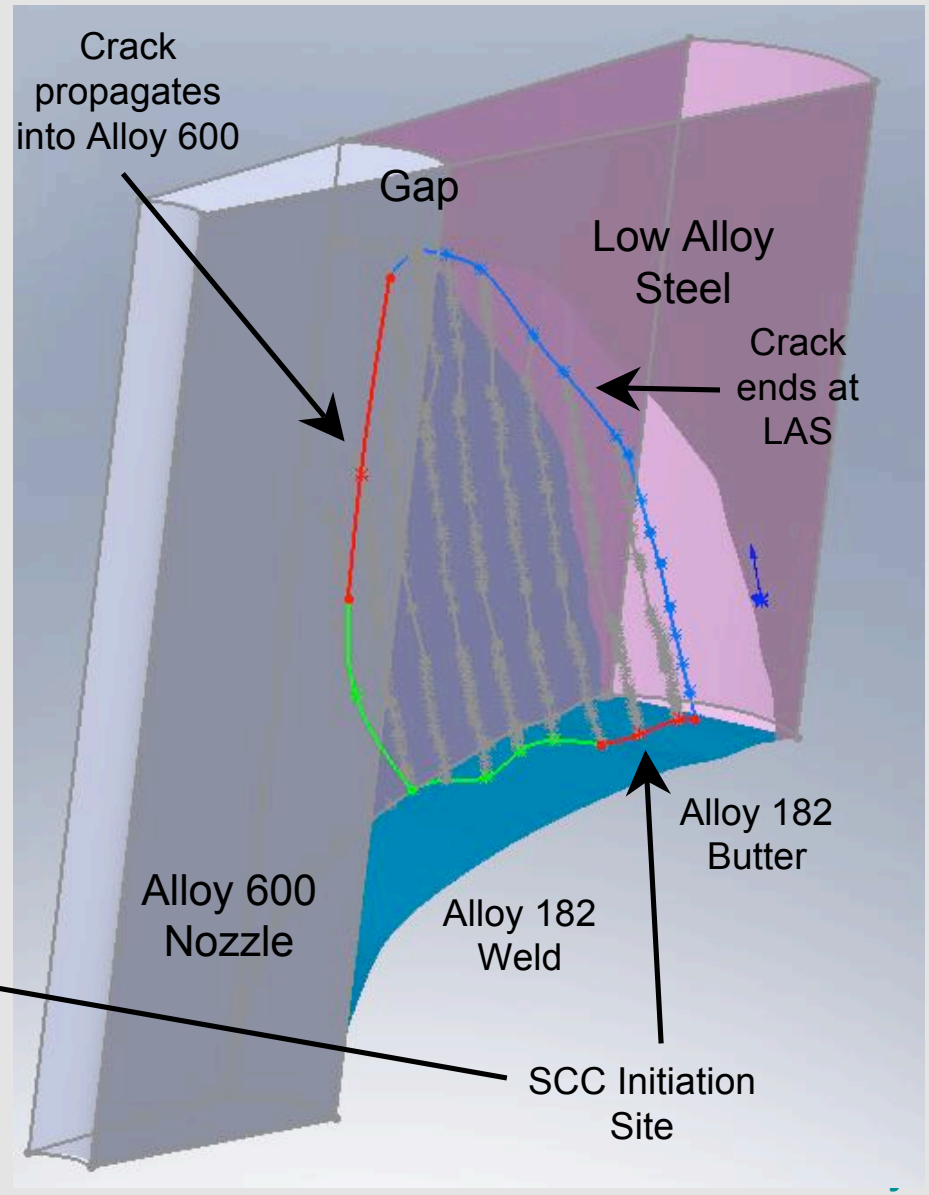
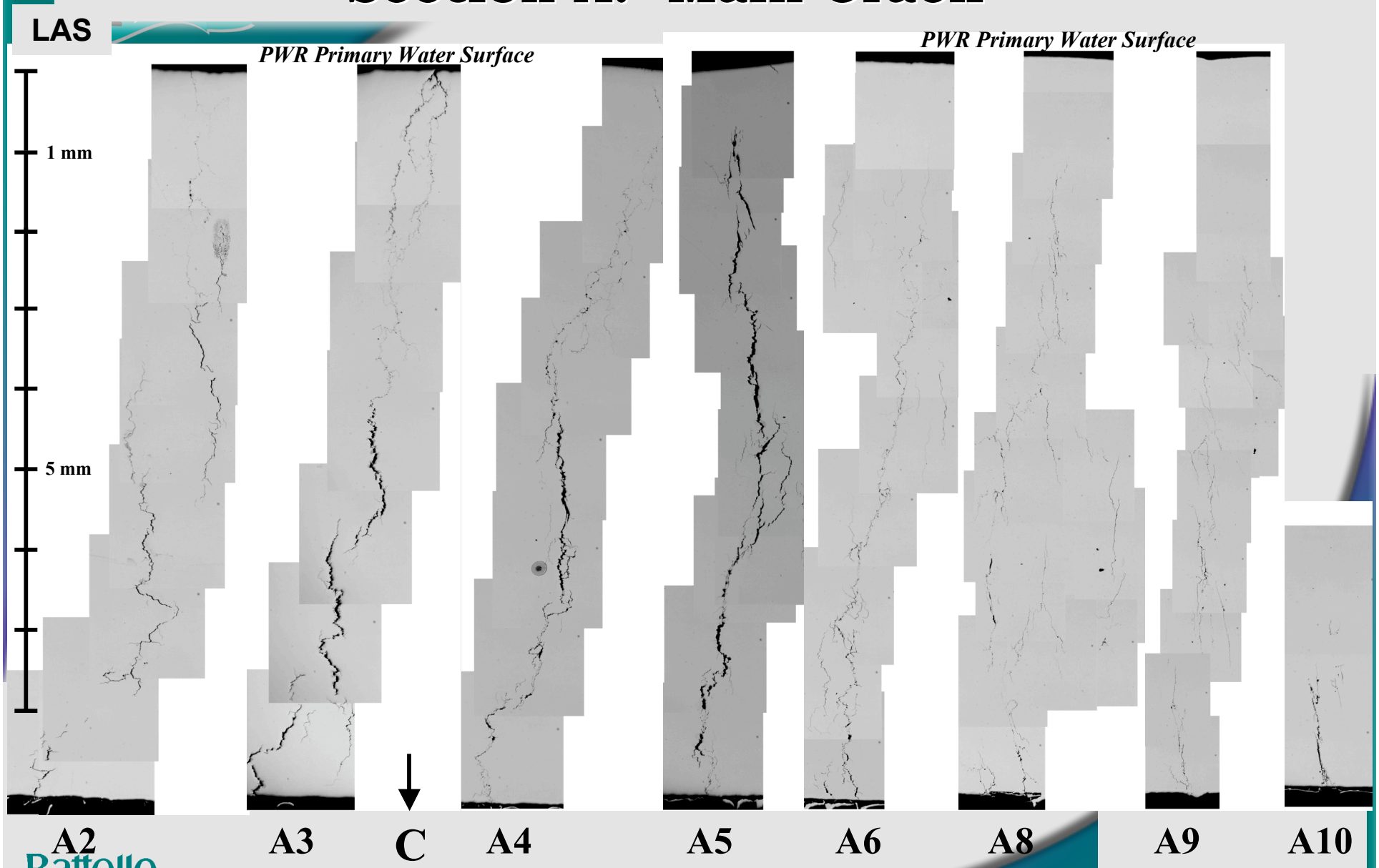
North Anna 2, Nozzle 31





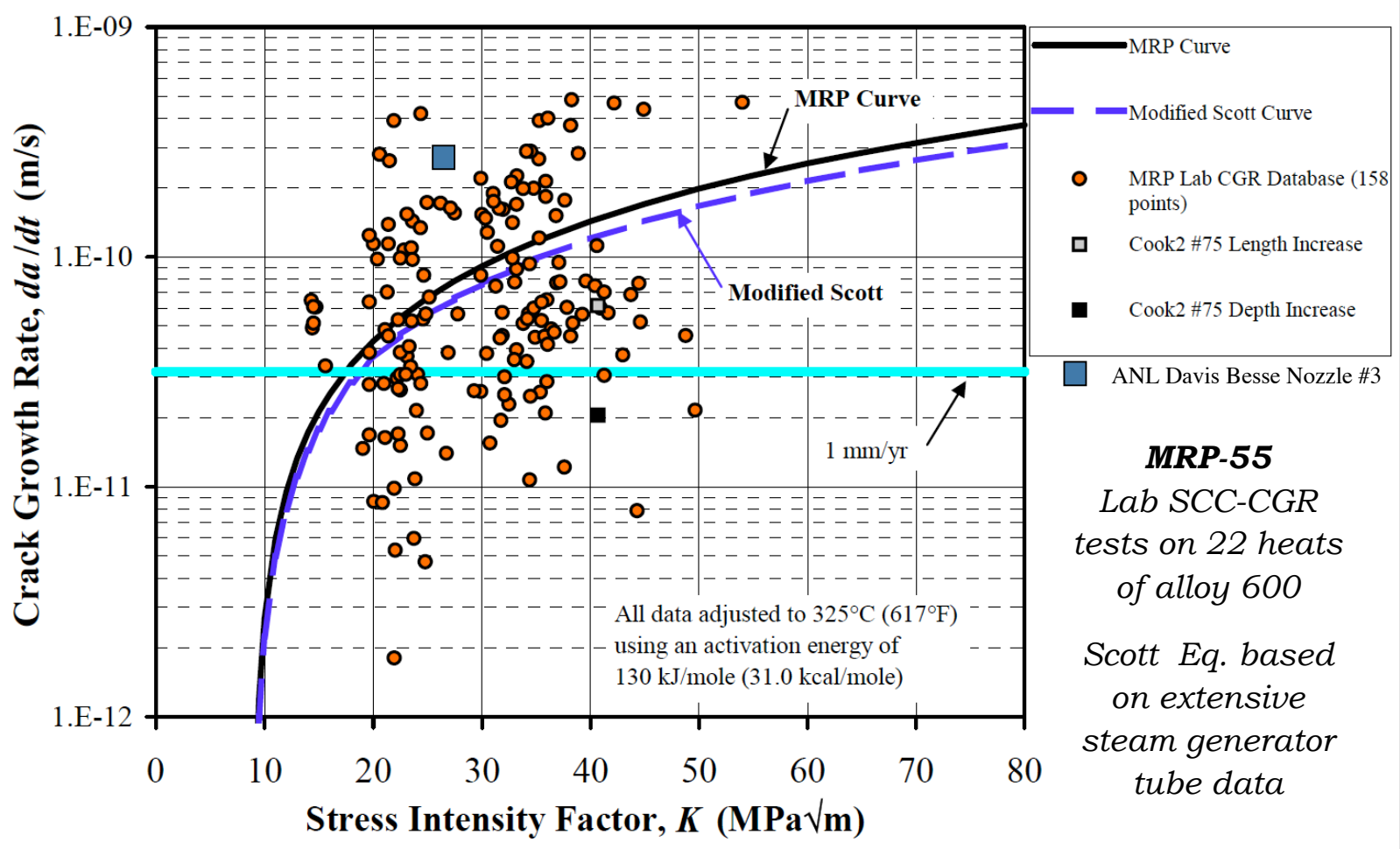


North Anna 2 Nozzle #31
Section A: Main Crack



Approximately 3 mm between slices, total length from A2 to A10 is ~26 mm

MRP Alloy 600 Data Base



MRP-55

Lab SCC-CGR tests on 22 heats of alloy 600

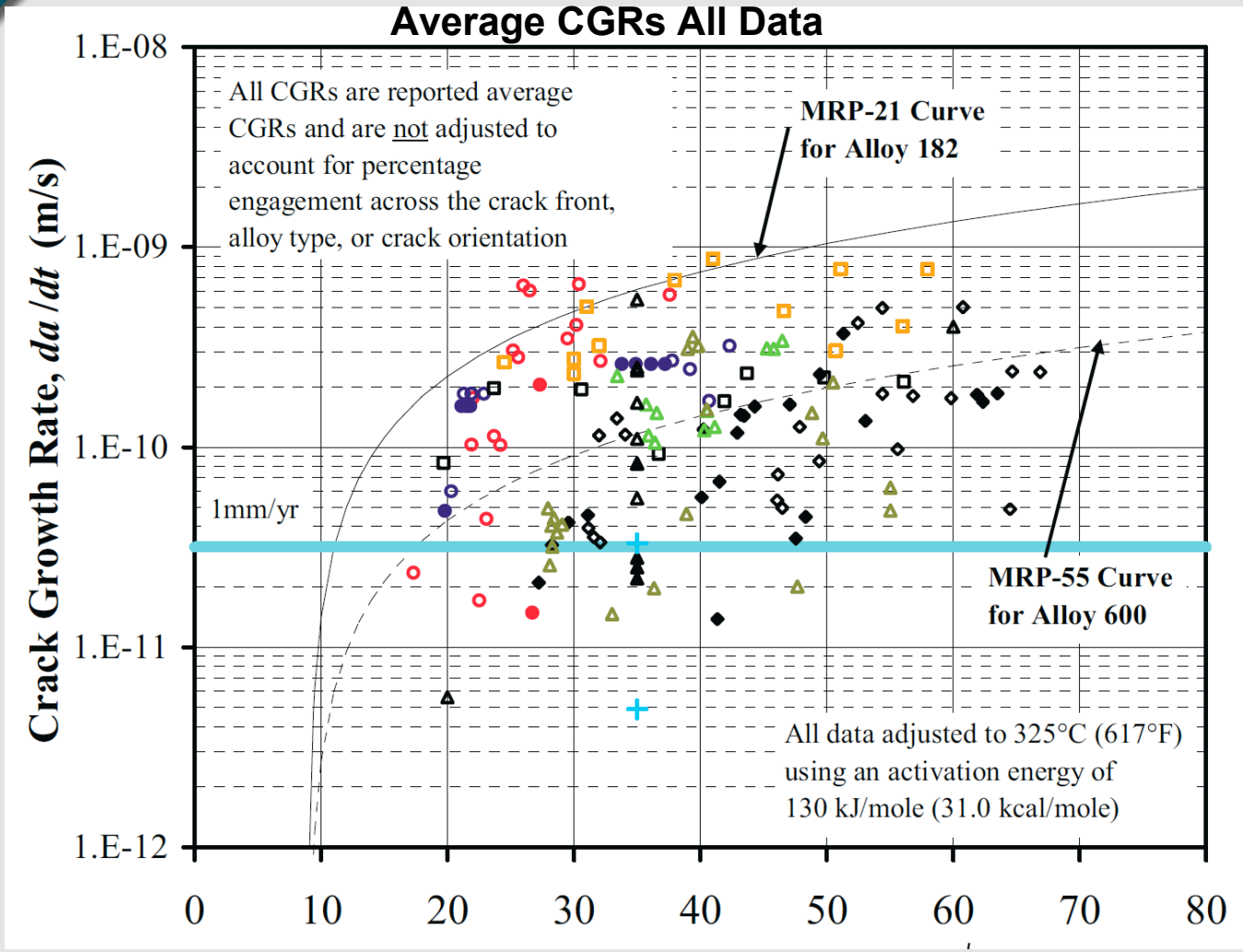
Scott Eq. based on extensive steam generator tube data

G. A. White, J. Hickling and L. K. Mathews, "Crack Growth Rates for Evaluating PWSCC of Thick Wall Alloy 600 Material," Proc. 11th Int. Conf. Environmental Degradation of Materials in Nuclear Power Systems, NACE, 2003, p. 166.

Alloy 600 SCC
Crack Growth Issues

- **Controlling Variables**
  - Strong Effect of Test Temperature (CGR ↑ as Temp ↑)
  - Strong Effect of Cold/Warm Work (Strength) on SCC
  - Maximum CGR at Ni/NiO ECP
  - Grain Boundary Carbides Influence SCC Resistance
- **Data Base Issues**
  - Good laboratory CGR data base for alloy 600 materials and excellent service SCC data foundation from PWR steam generator experience. Some concern that a K_{ISCC} threshold exists (assumed at 9 MPa√ m).
  - Limited quantitative CGR data for thick-wall PWR service materials, susceptible heats reflect high end of CGR distribution (>75%)? Indicate need for more tests on component materials removed from PWR service.

MRP Alloy 182/132/82 Data Base

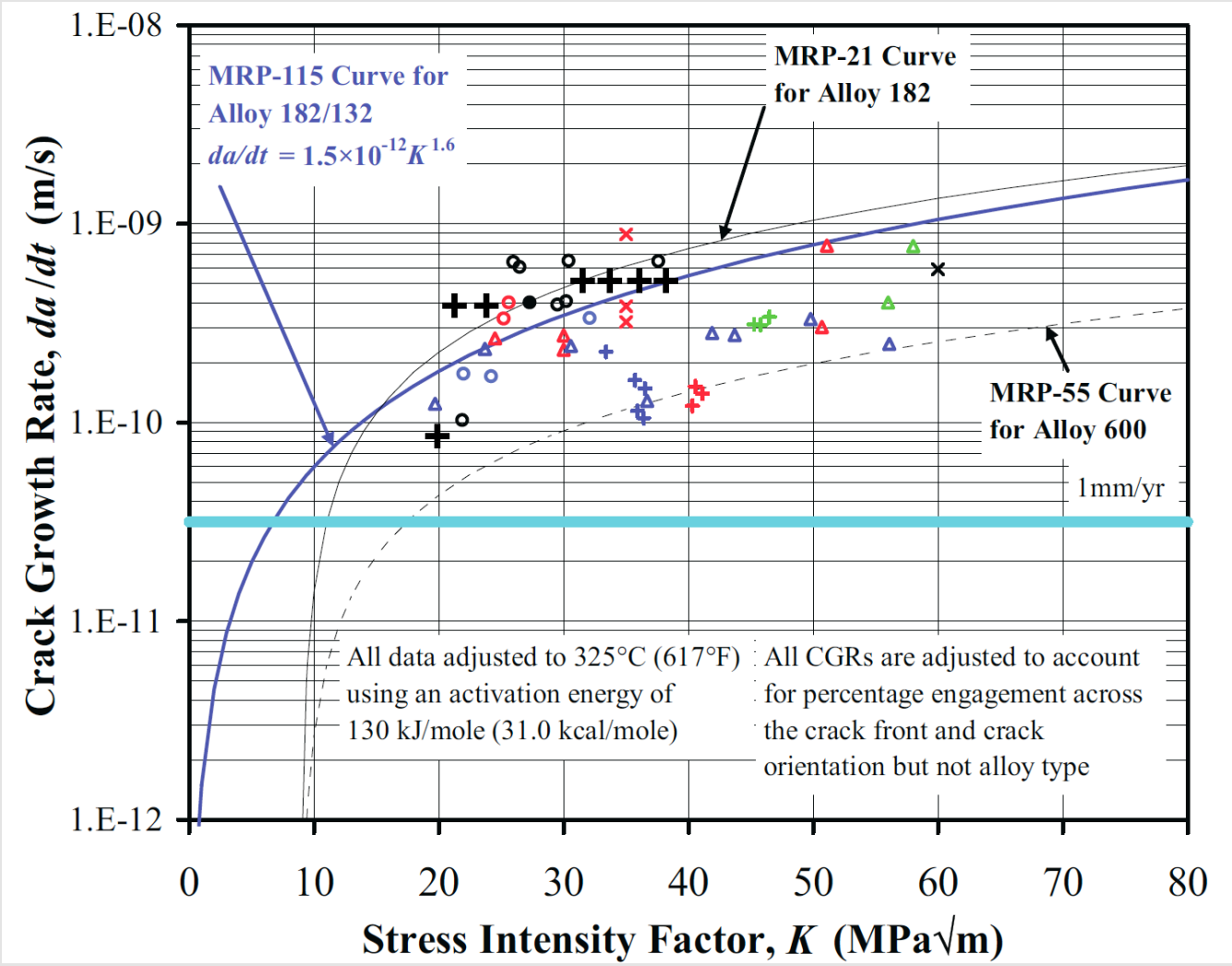


Alloy 82 consistently show lower CGRs than Alloy 182/132 by ~2-3X

G. A. White, N. S. Nordmann, J. Hickling and C. D. Harrington, "Development of Crack Growth Rate Disposition Curves for PWSCC of Alloy 82, 182 and 132 Weldments," Proc. 12th Int. Conf. Environmental Degradation of Materials in Nuclear Power Systems, TMS, 2005, p. 511.

MRP Alloy 182/132 Data Base

Adjusted CGRs - Crack Front Engagement and Orientation

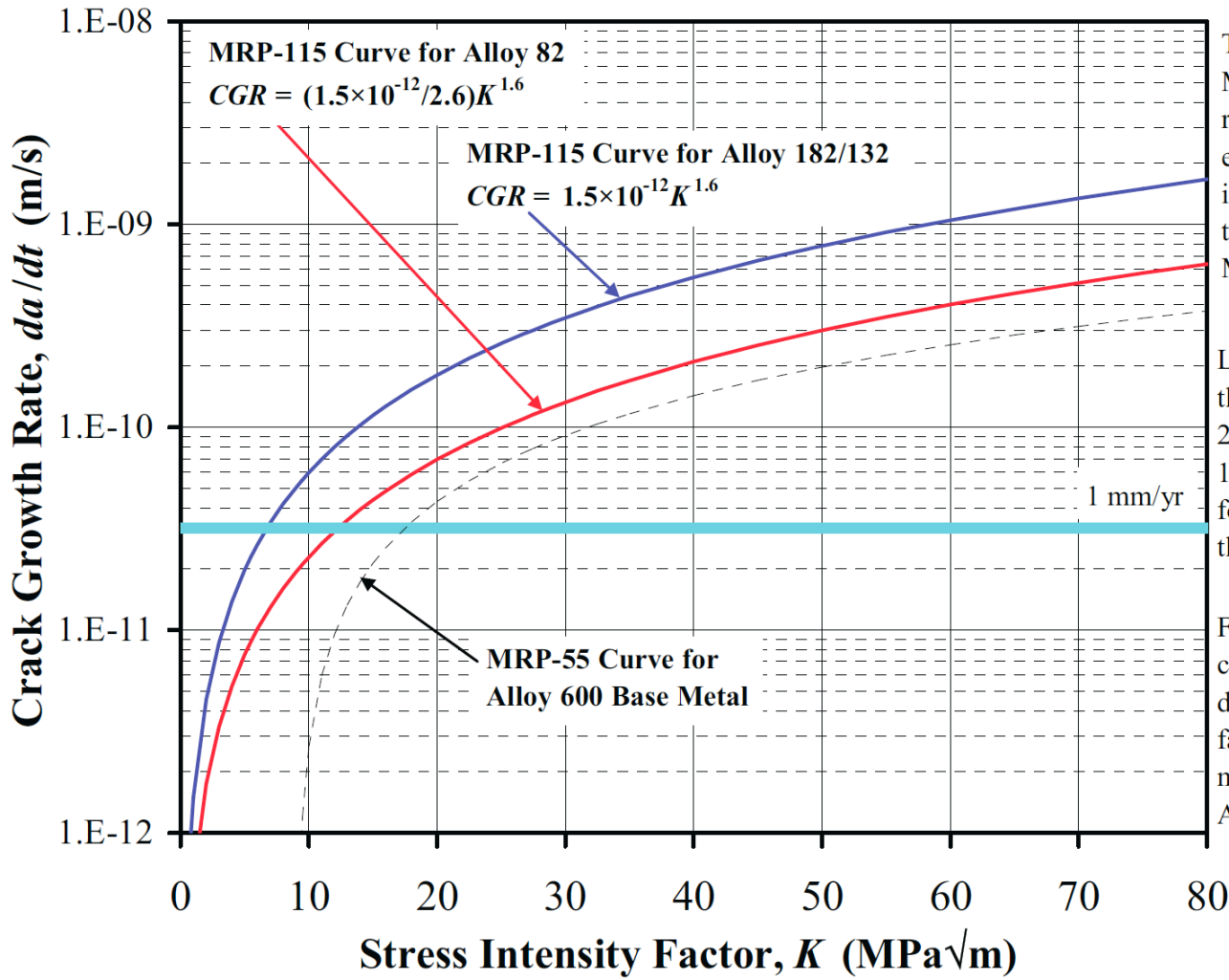


+ Alloy 182 Westinghouse Lab CGR Data VC Summer

Alloy 82 CGR Data not shown, again ~2-3X lower MRP-115 curve for alloy 82 is 2.6X lower than for alloy 182

G. A. White, N. S. Nordmann, J. Hickling and C. D. Harrington, "Development of Crack Growth Rate Disposition Curves for PWSCC of Alloy 82, 182 and 132 Weldments," Proc. 12th Int. Conf. Environmental Degradation of Materials in Nuclear Power Systems, TMS, 2005, p. 511.

Disposition Curves



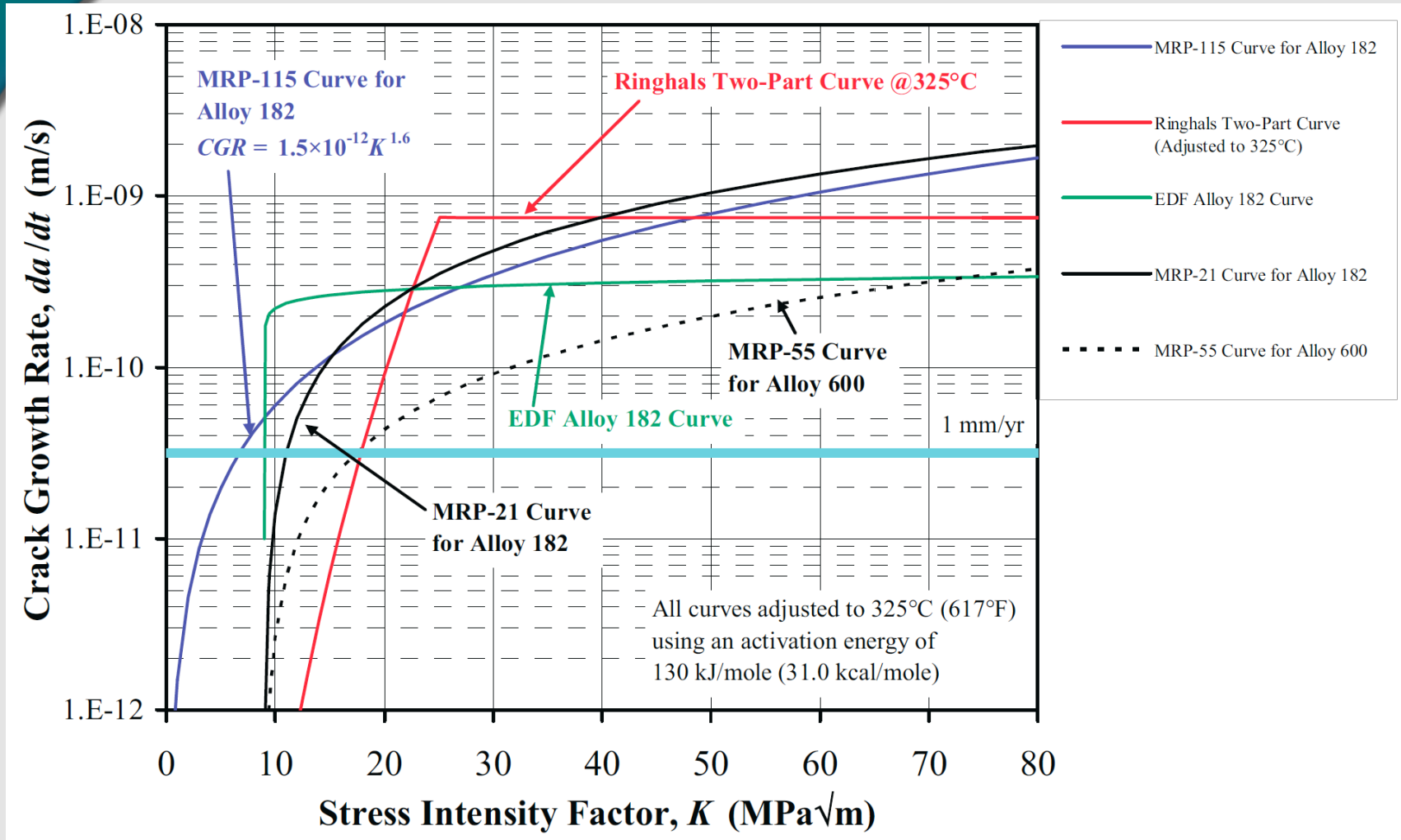
The reference temperature for the MRP curves is 325°C (617°F); the recommended thermal activation energy for temperature adjustment is 130 kJ/mole (31.0 kcal/mole), the same value recommended in MRP-55 for base metal.

Laboratory testing indicates that the CGR for Alloy 82 is on average 2.6 times lower than that for Alloy 182/132, so the MRP-115 curve for Alloy 82 is 2.6 times lower than the curve for Alloy 182/132.

For crack propagation that is clearly perpendicular to the dendrite solidification direction, a factor of 2.0 lowering the CGR may be applied to the curves for Alloy 182 (or 132) and Alloy 82.

G. A. White, N. S. Nordmann, J. Hickling and C. D. Harrington, "Development of Crack Growth Rate Disposition Curves for PWSCC of Alloy 82, 182 and 132 Weldments," Proc. 12th Int. Conf. Environmental Degradation of Materials in Nuclear Power Systems, TMS, 2005, p. 511.

Disposition Curves

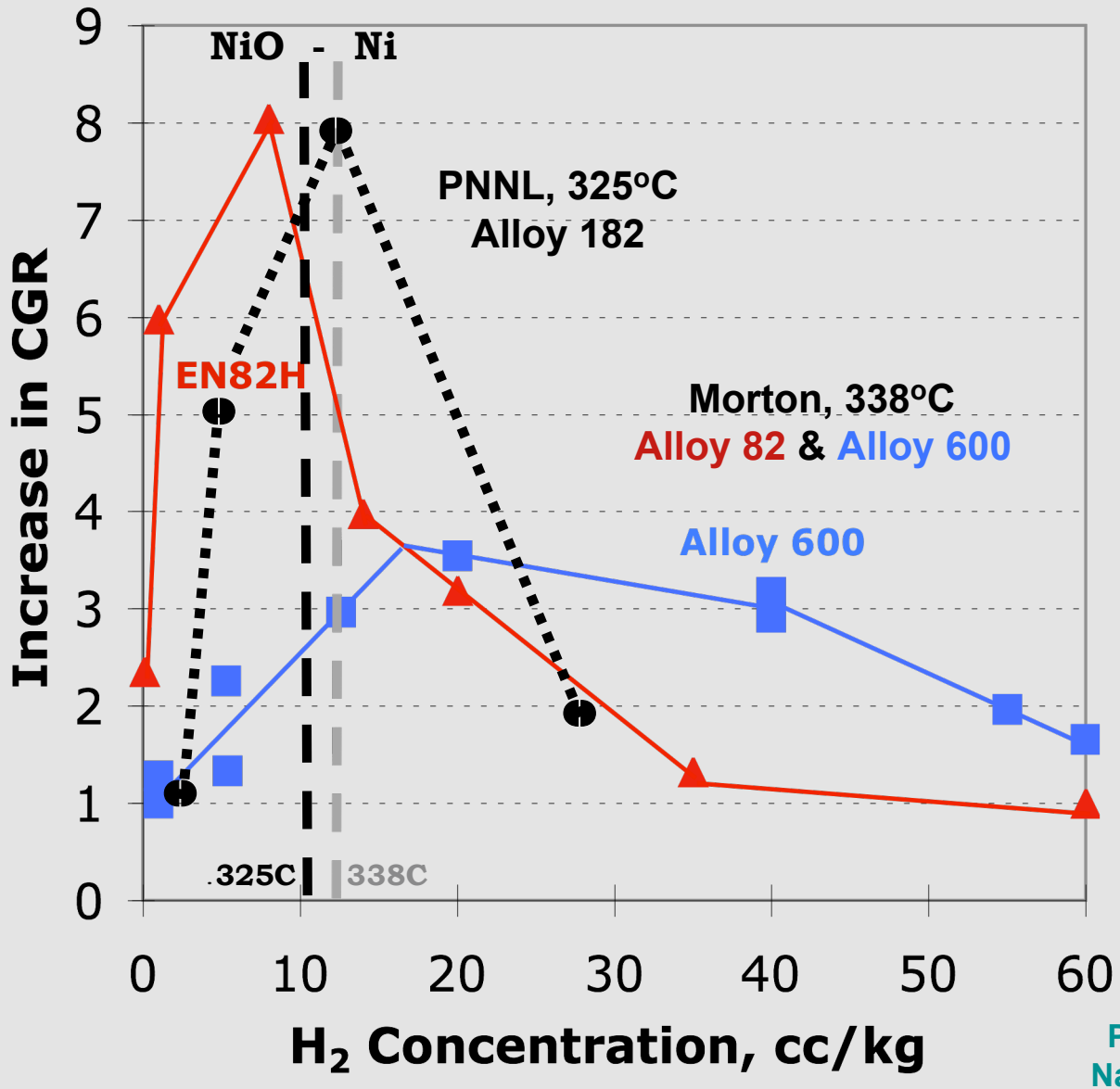


G. A. White, N. S. Nordmann, J. Hickling and C. D. Harrington, "Development of Crack Growth Rate Disposition Curves for PWSCC of Alloy 82, 182 and 132 Weldments," Proc. 12th Int. Conf. Environmental Degradation of Materials in Nuclear Power Systems, TMS, 2005, p. 511.

Alloy 182/82 SCC Crack Growth Issues

- **Controlling Variables**
  - Strong Effect of Test Temperature (CGR↑ as Temp↑)
  - Strong Effect of Cold/Warm Work (Strength) on SCC
  - Strong Effect of ECP (H_2 concentration), maximum CGR at Ni/NiO ECP
  - Strong Effect of Orientation (Dendrite/Grain Structure)
- **Data Base Issues**
  - Reasonable laboratory CGR data base for alloy 182/82, but complex material issues due to weld variability. Some additional evaluation need on H_2/ECP, temperature, materials interrelations on CGRs.
  - Limited quantitative data for thick-wall PWR service materials, susceptible heats could reflect high end of CGR distribution? Indicate need for additional tests on component materials removed from PWR service.

H_2 Effects on SCC Crack-Growth



PNNL FY08 Research Plans: Alloy 600/182

- **Alloy 600**
  - Complete tests examining H_2/ECP effects on highly cold worked (30%) alloy 600.
  - Characterizing microstructure, SCC cracks and crack tips that have propagated into North Anna 2 nozzle 31.
- **Alloy 182 Weld Metal**
  - Complete tests examining H_2/ECP effects on "SCC-susceptible alloy 182 weld metal from EdF.
  - Machining samples from non-cracked nozzle weld from North Anna 2 material for CGR samples from alloy 182 butter and in main alloy 182 J-groove weld.
  - Characterizing microstructure, SCC cracks and crack tips in North Anna 2 nozzle 31, both in alloy 182 butter passes and in the main J-groove weld. In addition, pits are being characterized that have developed at low alloy steel interface where SCC cracks end.

Presentation Topics

- **Laboratory SCC Crack-Growth Testing**
- **Crack-Growth Rates for Alloy 690/152/52**
  - Current Alloy 690/152/52 Testing and Data Summary
  - Potential Issue for 1D-Rolled Alloy 690
  - Questions, Concerns, Issues and Research Needs
- **Crack-Growth Rates for Alloy 600/182/82**
  - Alloy 600 Data and Disposition Curve
  - Alloy 182/82 Data and Disposition Curves
  - Questions, Concerns, Issues and Research Needs
- **SCC Crack-Growth Testing Issues**
  - System Requirements and Test Approach
  - Problems/Issues for SCC-Resistant Materials

SCC Crack Growth Testing Issues

Many experimental, materials, enviromental and interpretation elements necessary for effective measurement/analysis of SCC:

1. *Successful SCC testing requires a broad knowledge of metallurgy, mechanics, chemistry, electrochemistry, and physical measurements - excellence not in a few areas, but in all*
2. *Test methodology important - transitioning, unloading, K/size, crack front evenness, dcpd resolution, test management*
3. *Material (heat, processing, homogeneity, heat treatment, inclusion/carbides, cold work/HAZ, orientation, …) and environment (temperature, pH, purity, …) variations important*
4. *Interpretation important – uneven crack fronts & data correction, use of avg vs. max CGR, K correction, data reproducibility…*

Reproducibility of SCC CGR Data Depends on Many Items Including Test System Capabilities:

1 – Loading stability, dK/da correction, high-to-low frequency cycling, cycle + hold, constant displacement control, …

2 – DCPD crack length measurement *resolution & accuracy (need at least <10 μm, <2 μm for SCC resistant alloys)*

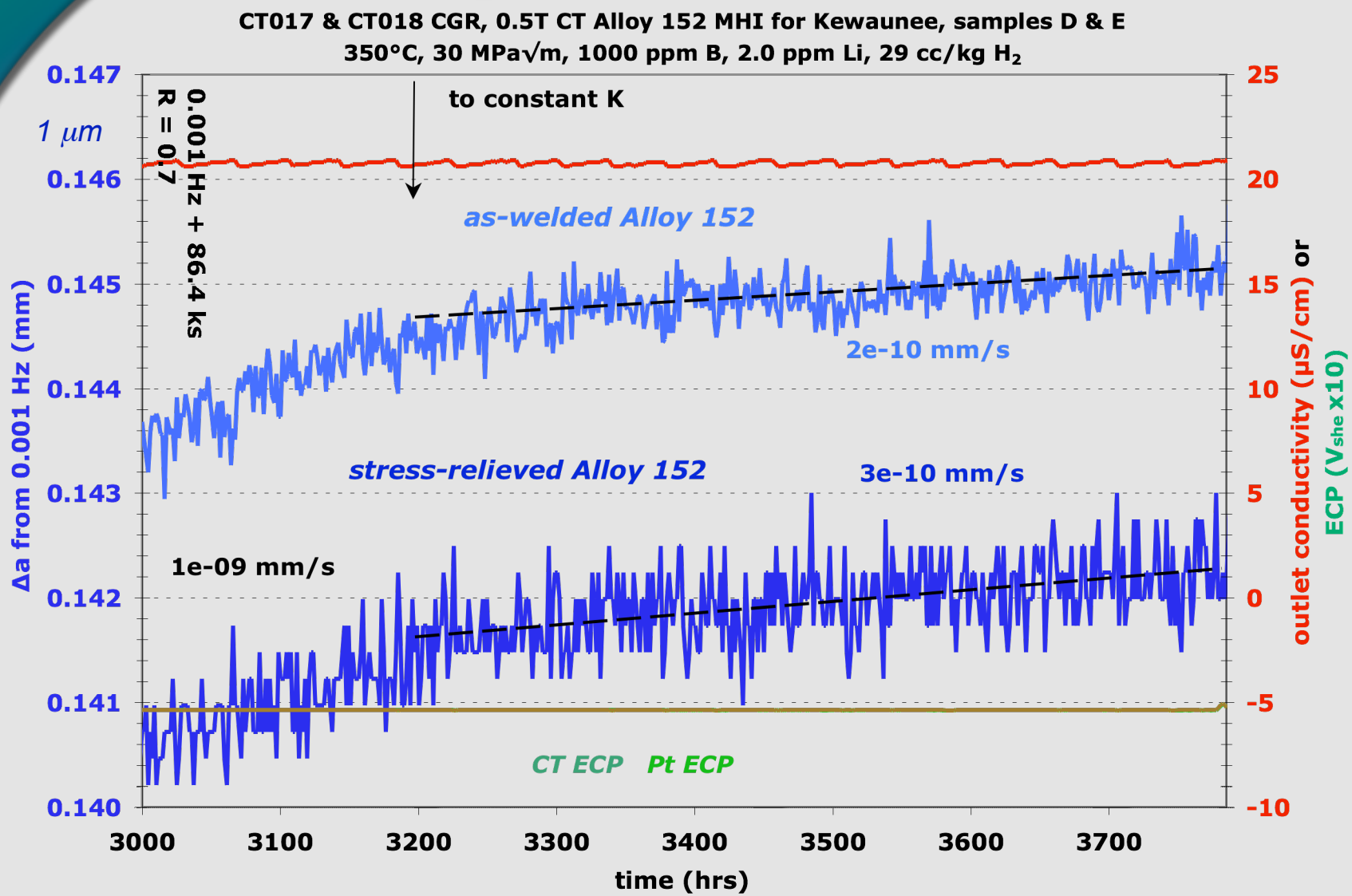
3 – Temperature (<0.2°C fluctuation), water pressure, seal friction and water chemistry (<0.1 μS/cm outlet) control

4 – Reference electrode accuracy & reliability

5 – Ability to make changes "on-the-fly", e.g., in temperature, H_2 concentration, pH/B/Li/impurities, …

6 – Maintain continuous operation and stability *of all test conditions* ***over long times*** *(e.g., >12 months)*

Alloy 152: Constant K



Stable system and high-resolution DCPD capable of measuring extremely slow, stable crack growth (μm dimensions) at constant K over many months

Reproducibility of SCC CGR Data Depends on Many Items Including Test Approach Aspects:

1 – Wise management of testing and specimen response

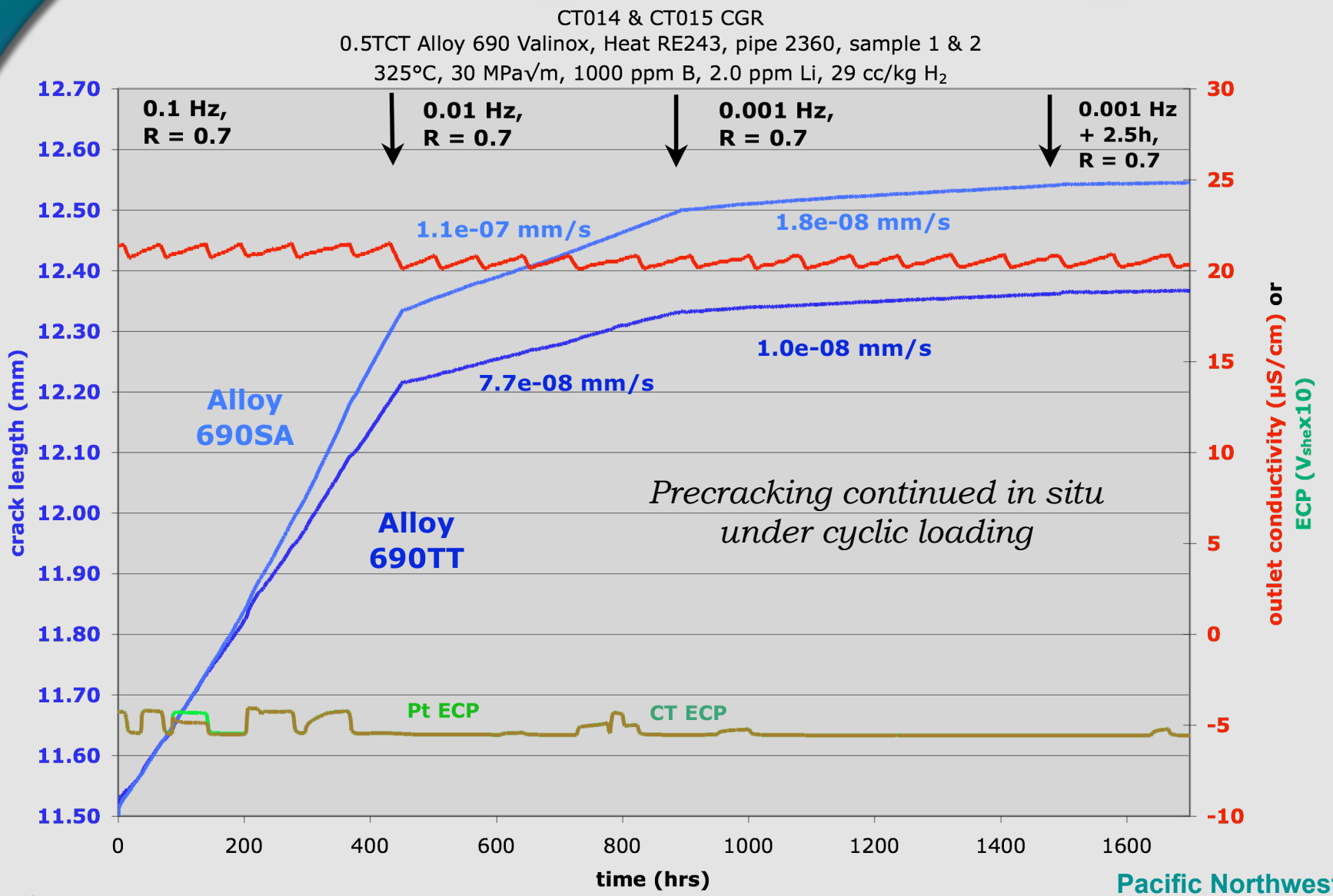
***2 – Transition from TG fatigue precrack to IGSCC**, e.g., use decreasing cyclic frequencies, increasing R values and increasing hold times at K_{max}*

3 – Maintain straight crack front and minimize "fingers" of SCC growth as possible

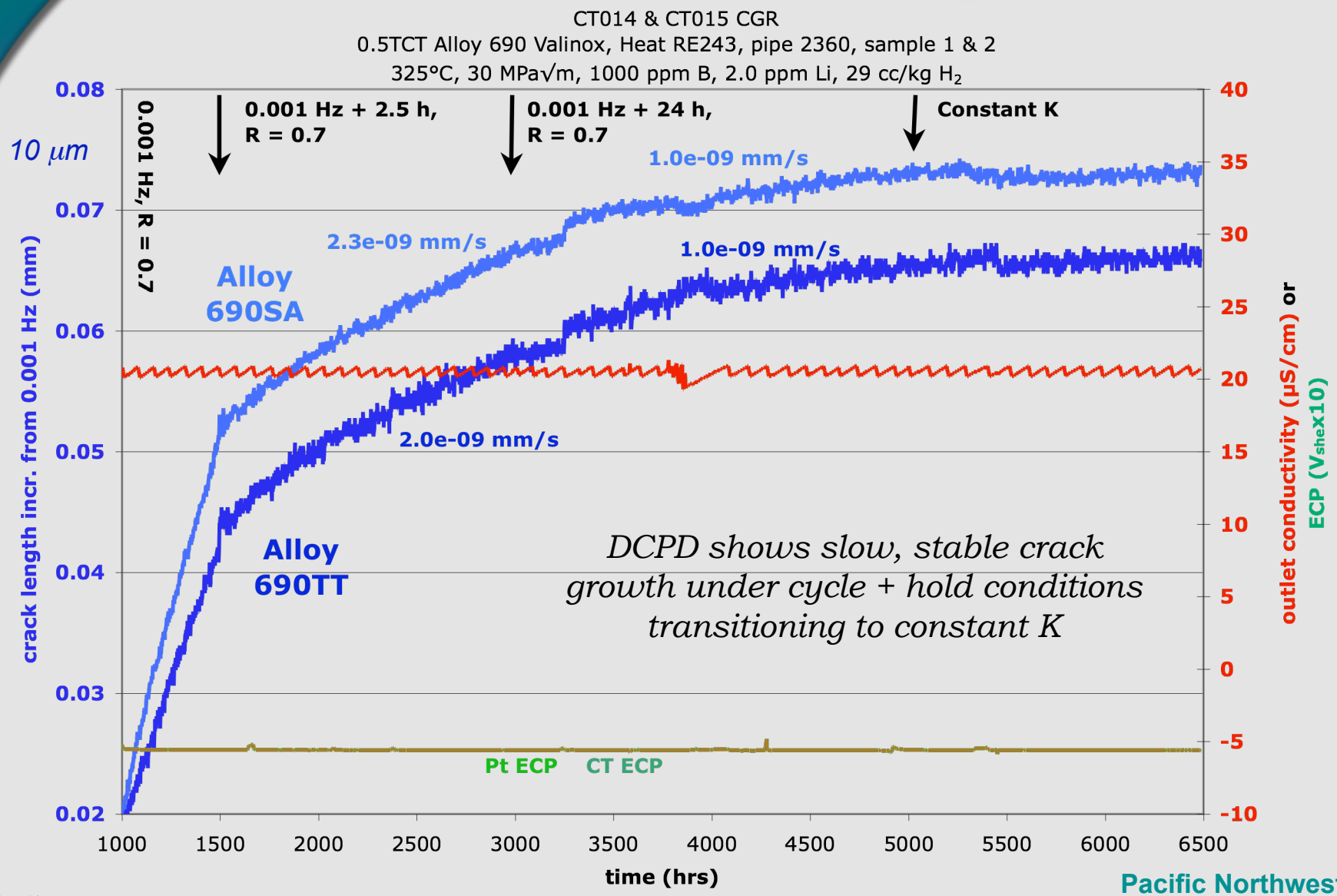
4 – Repeat crack growth rate measurements for key conditions after different crack extensions to sample different microstructural regions

5 – Intelligent post-test crack length correction to obtain accurate K levels and average/maximum growth rates

Alloy 690 (TT/SA) Precracking and Transitioning



Alloy 690 (TT/SA) Crack Transitioning



Battelle

Pacific Northwest National Laboratory

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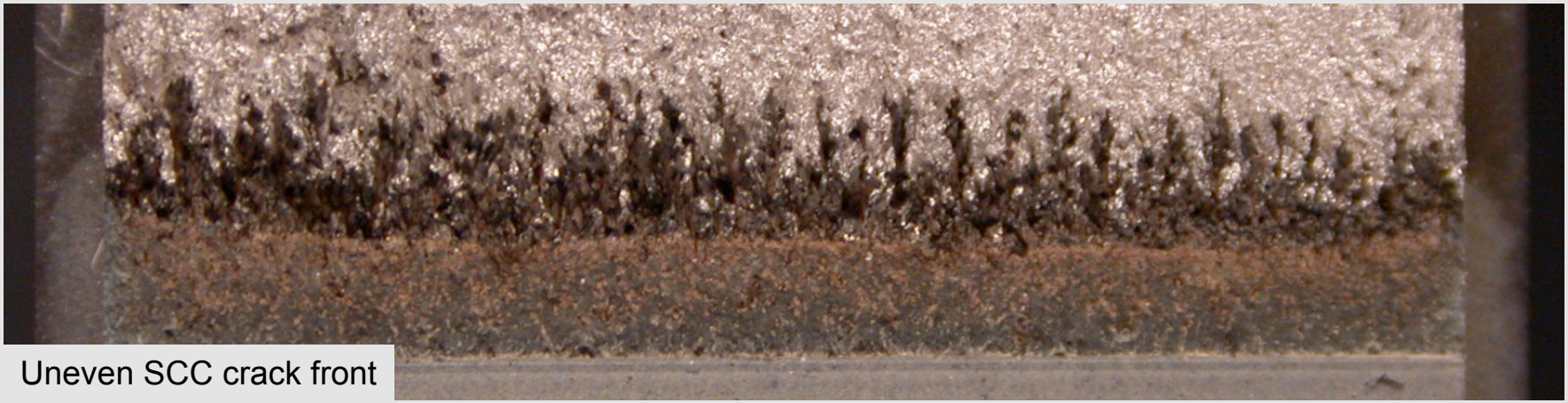
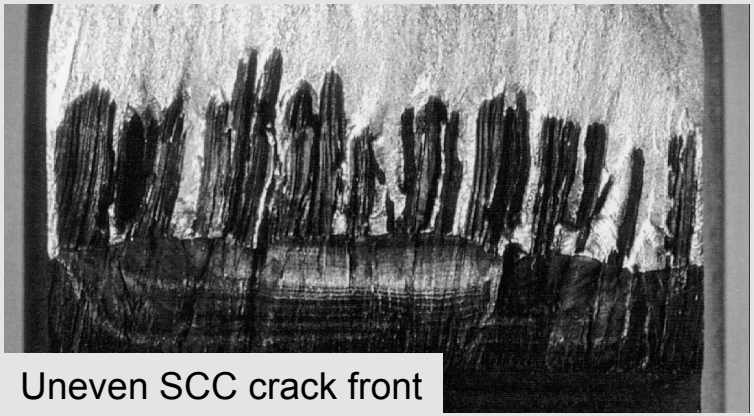
TG Fatigue Precracking and IGSCC Transitioning

TG fatigue cracks poorly simulate lab or field IGSCC. Morphology change, plastic zone, crack front pinning issues. Attempt to transition to IGSCC during low frequency cycling + hold times.



Uneven SCC crack front



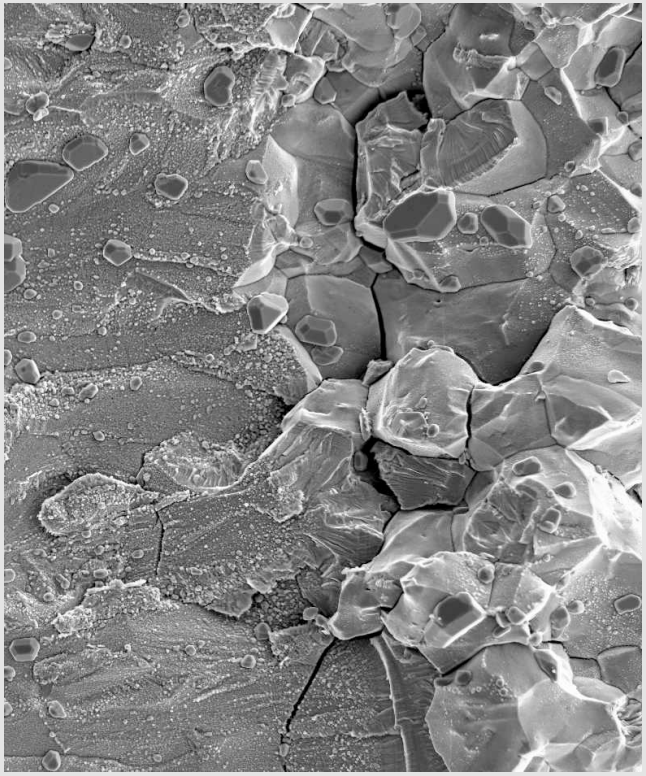
Uneven SCC crack front

Uneven fatigue crack front and local SCC

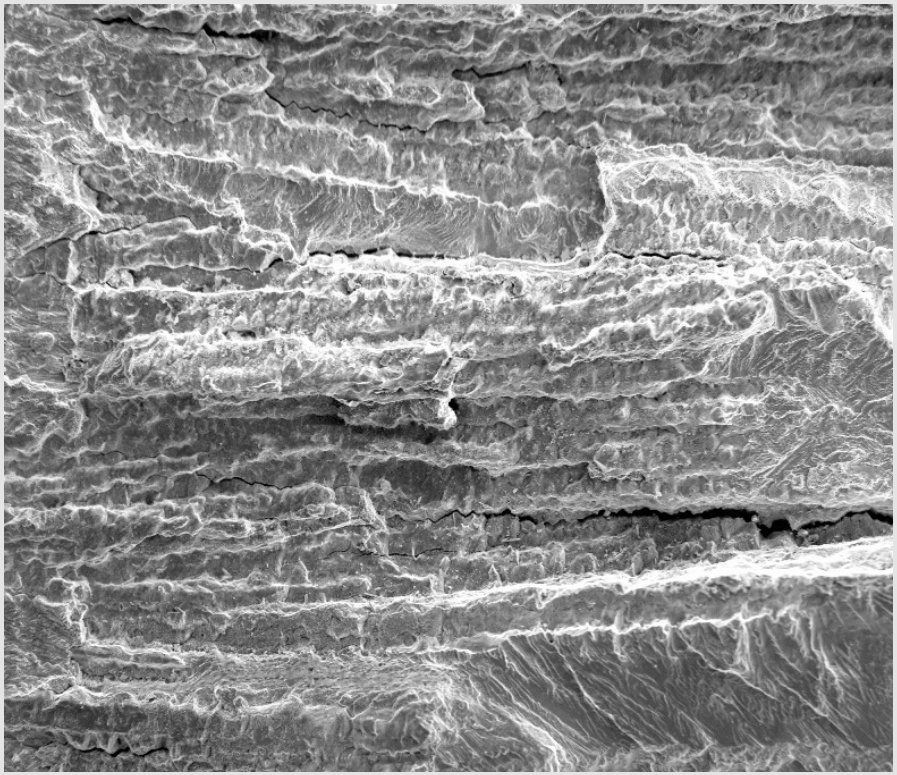
Figures from P. L. Andresen

IGSCC Transitioning and Reproducible Crack Growth Data

Best results obtained for crack front fully engaged and transitioned to IGSCC. Complex microstructures such as for weld metals can make fully engagement difficult.



TG fatigue crack - IGSCC in wrought 304SS



IGSCC along dendritic boundaries in alloy 182 weld metal

Reproducibility of SCC CGR Data Depends on Many Items Including Test Approach Aspects:

1 – Wise management of testing and specimen response

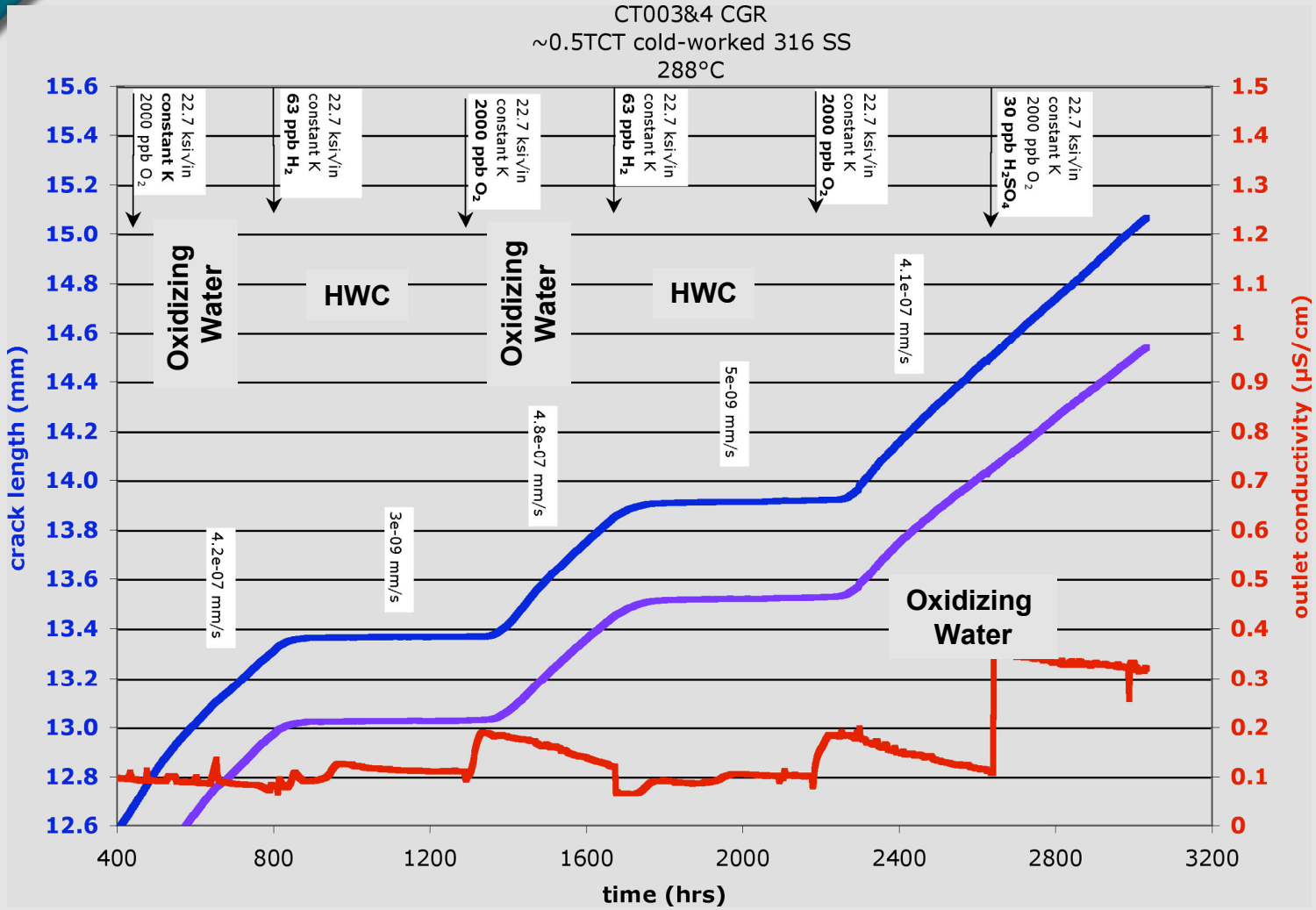
2 – Transition from TG fatigue precrack to IGSCC, e.g., use decreasing cyclic frequencies, increasing R values and increasing hold times at K_{max}

3 – Maintain straight crack front and minimize "fingers" of SCC growth as possible

***4 – Repeat crack growth rate measurements for key conditions** after different crack extensions to sample different microstructural regions*

5 – Intelligent post-test crack length correction to obtain accurate K levels and average/maximum growth rates

CGR Test Approach



Demonstrate data reproducibility by repeating key conditions during single test, e.g., O_2 (4-5x10^{-7} mm/s) versus H_2 (3-5x10^{-9} mm/s)

IG Transitioning and SCC Test Management

Criterion for success is pragmatic: Does it work?

SCC resistant materials are much harder to transition, IG path has low susceptibility similar to TG.

1 – Avoid large changes in K_{max} to minimize blunting
2 – Use higher R to simulate constant K, but $R \geq 0.8$ too often fails to accelerate SCC
3 – Use frequency and hold time at K_{max} to enhance EAC.
4 – Must monitor material response on-line for real-time assessment of transitioning; a fixed formula of loading and time rarely works for SCC resistant materials.

Chemistry Disconnects in Comparing Data

Large number of variables to monitor and control during a test, often not reported, creates data uncertainties.

1 – H_2 control and stability; H_2 vs. test temp; normalizing data.
2 – Test temperature: coordinated H_2; issues with temperature activation energies for base metal, weld metal, 1D CW?
3 – Impurities in water and H_3BO_3 , esp. in static autoclaves.
4 – Possible concern for autoclave system materials and transport of metal cations to the specimen.
5 – Test start up and interruption may perturbate data, but this is more of an issue in crack initiation experiments.

Lab control of PWR primary water chemistry is a smaller issue than BWR, but some concerns remain.

Material Disconnects in Comparing Data

1 – Heat-to-heat variability, including cleanliness

2 – Homogenization during early processing (vs. banding….)

3 – Anneal & thermal treatment (e.g., grain boundary segregation and precipitation)

4 – Residual or intentional cold work

5 – Welding procedures – dilution, residual stress & strain, heat input, interpass temperature…

6 – Processing: cold work per pass, 1-D vs. 2-D (cross-rolling or forging) or 3-D, temperature of working…

7 – Specimen orientation vs. welding, rolling, HAZ alignment, etc.

Test materials should be exchanged among testing laboratories, esp. when unusual behavior is observed

Interpretation Disconnects in Comparing Data

1 – Material differences/variability/orientation unknowns

2 – Initial pre-cracking, transitioning and test management

3 – Constant K testing, effect of periodic unloading

4 – Test duration and Δa, crack re-activation, test management

- *constant displacement vs. active load, esp. at lower K*

5 – Crack non-uniformity posts interpretation challenges:

- *recalcitrant areas can retard overall SCC along crack front*
- *rapid growth along dendrites increases K & allow 2D growth*

6 – Active test management much better than "load-and-hold"

7 – Post-test correction – effect on CGR and K (avg vs max…), can be difficult to accurately correct for test stages

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