SCC Crack Growth Testing and Characterization of Alloy 690 and its Weld Metals at PNNL

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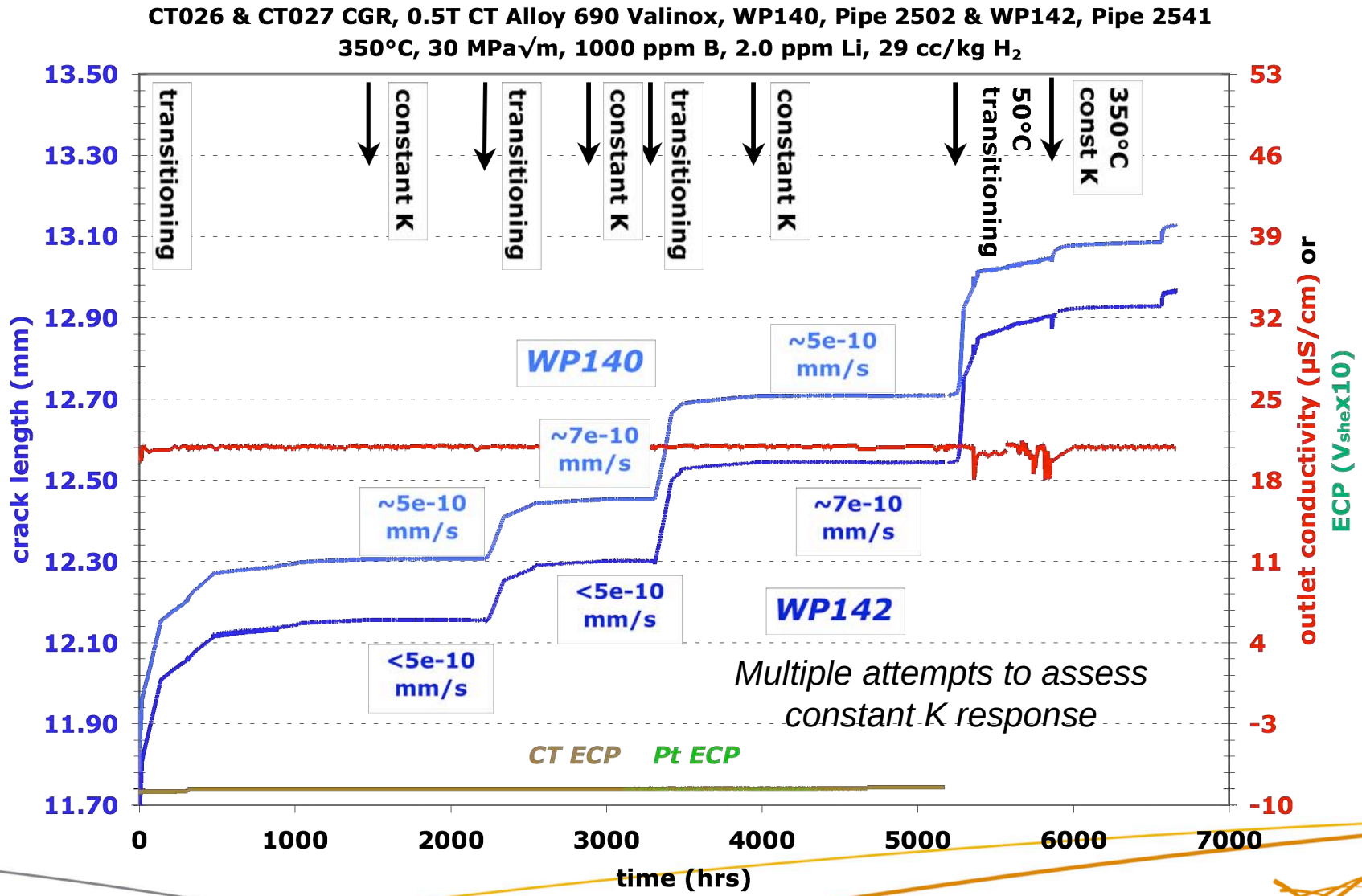
Crack Growth Testing Methodology

- Alloy 690/152/52 are very resistant to stress corrosion cracking in nearly all material conditions, has been very difficult to obtain stable crack growth.
- As a means to assess constant K growth rates and obtain maximum SCC crack front engagement, different transitioning methods have been investigated and are routinely used.
  - Varied R from 0.5 to 0.7
  - Varied hold time
  - Varied length of time at each step during transitioning
  - Varied cyclic loading waveform rise time and fall time
- Transition to constant K several times in each test.
- If possible, examine crack growth response at two K levels.

Alloy 690 CRDM Heats WP140, WP142 SCC Test Overview



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Alloy 690 CRDM Materials

Alloy 690 CRDM tubing obtained from Valinox Nucléaire

- Six different heats, seven tubes
- Several different tubing dimensions

Alloy 690 CRDM Compositions

- RE243: 10.4Fe, 28.9Cr, 0.02C, 0.31Mn, 0.35Si, 0.14Al, 0.23Ti, Bal Ni
- WP142: 10.5Fe, 29.0Cr, 0.02C, 0.31Mn, 0.35Si, 0.18Al, 0.27Ti, Bal Ni
- WP140: 10.4Fe, 29.0Cr, 0.03C, 0.31Mn, 0.33Si, 0.18Al, 0.30Ti, Bal Ni

Alloy 690 Plate Materials

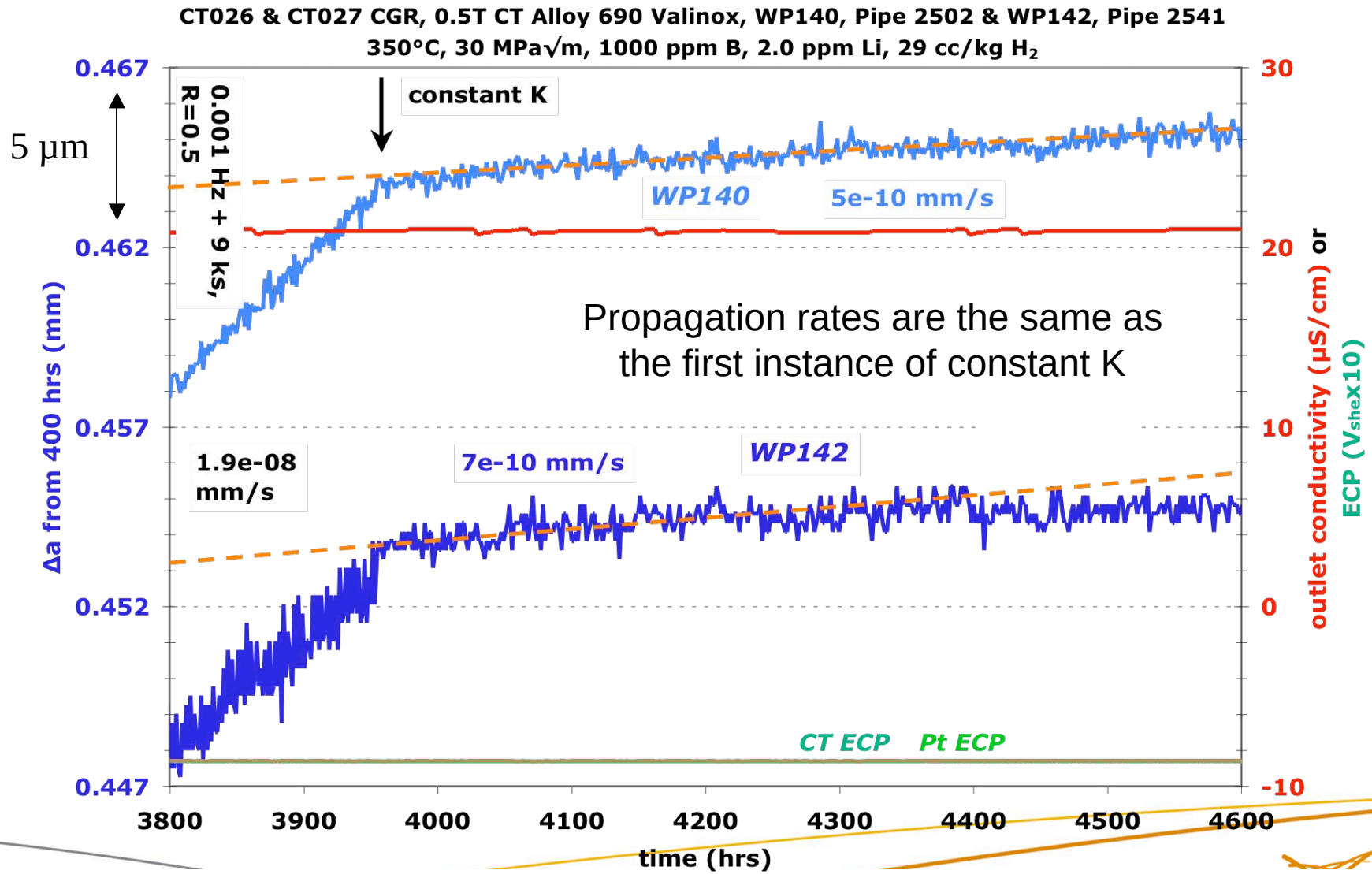
- EPRI NX8625HG21 (supplied by Al McIlree)
- ANL NX3297HK12 (supplied by Bogdan Alexandreanu)
- GEG B25K (supplied by Peter Andresen)

Alloy 690 Crack Growth Tests in Simulated PWR Water at PNNL

Alloy 690 Testing - 8 specimens complete, 2 on-going

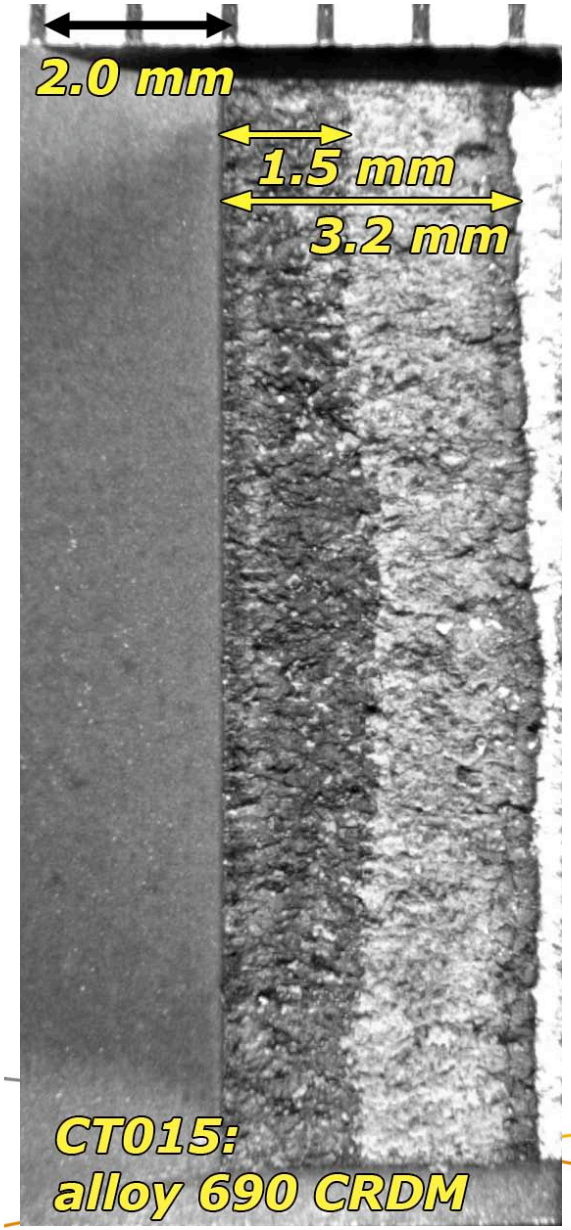
- Valinox CRDM Heat RE243: specimen CT014 in a carbide-modified (CM) condition, specimen CT015 in the as-received thermally treated (TT) condition
- Valinox CRDM Heat: specimen CT026 is heat WP142 in TT condition and specimen CT027 is heat WP140 in TT condition
- Valinox CRDM Heat RE243, specimen CT019 in TT + 17% cold-rolled (CR) in S-L orientation and specimen CT020 in CM + 17% CR (S-L) condition
- Valinox CRDM Heat RE243, specimen CT022 in a TT + 30% CR (T-L) condition and specimen CT023 in CM + 30% CR (T-L) condition
- Cold-Rolled Plate: specimen CT036 - ANL heat in 26% CR (S-L) condition and specimen CT037 - GEG heat in 20% CR (S-L) condition; on-going

Alloy 690 TT Heats WP140, WP142 Constant K

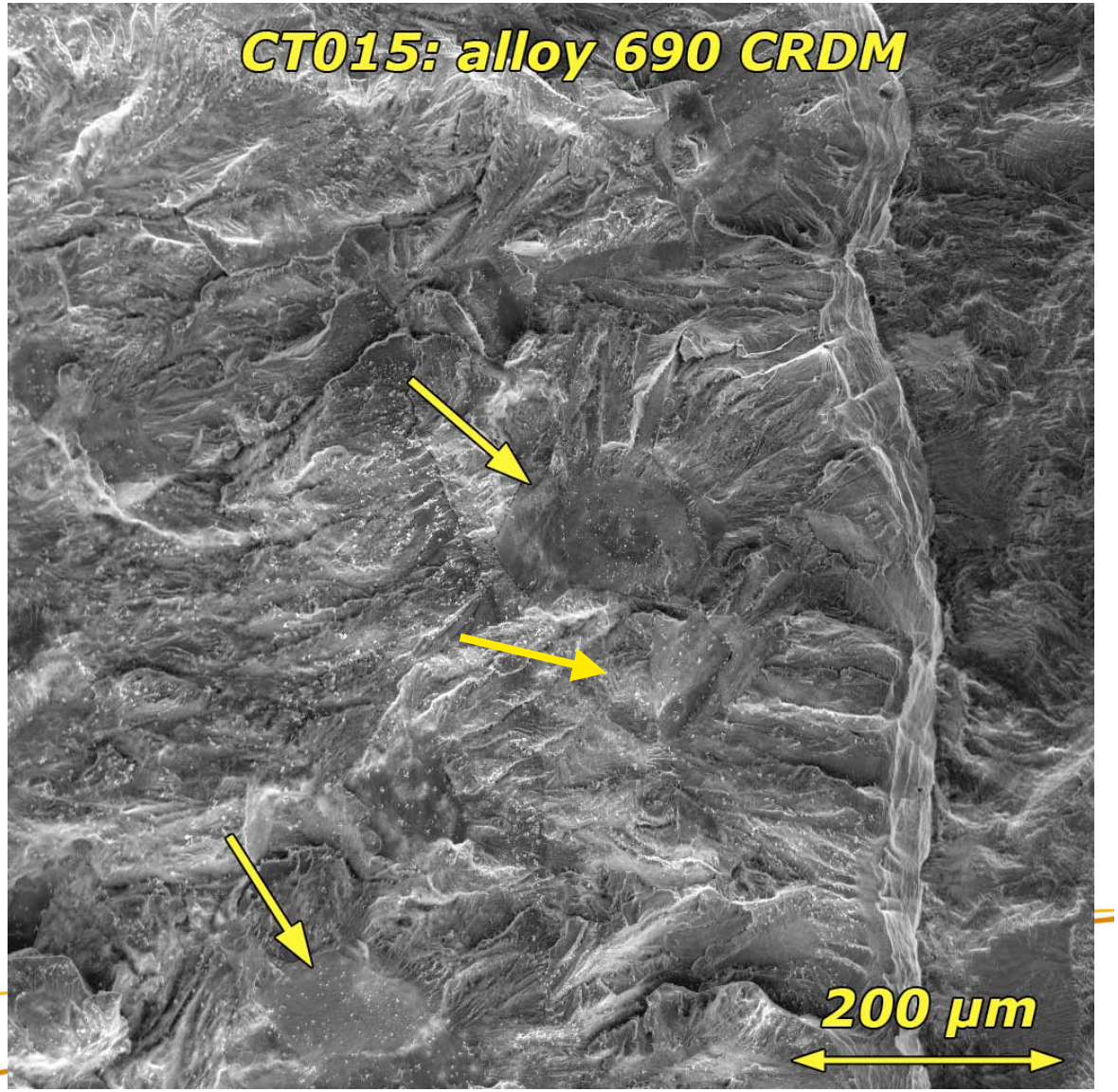


Similar very low growth rates measured after four separate transitioning attempts

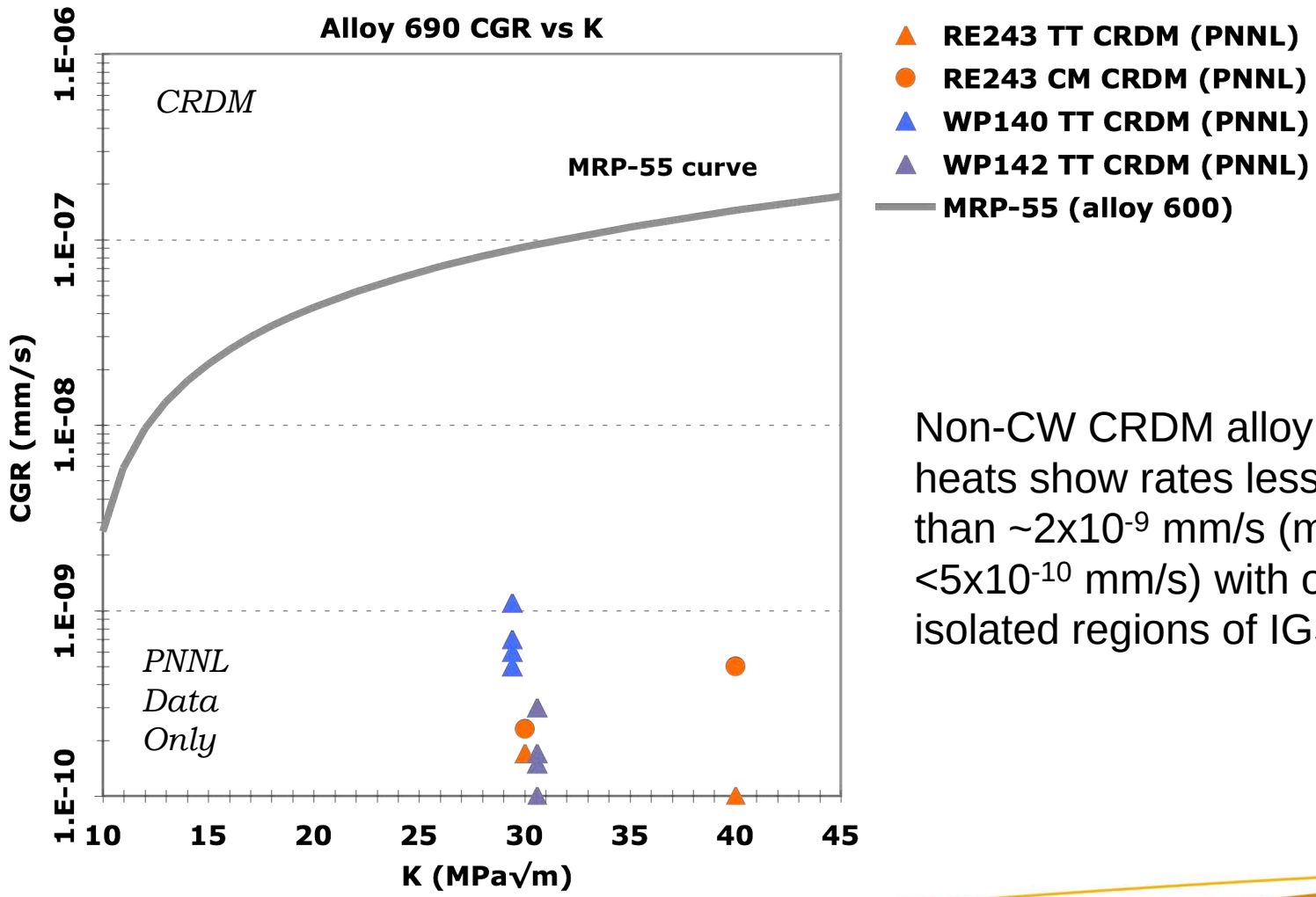
Alloy 690 Heat RE243 (TT/CM) 350°C Crack Growth Surface



IG cracking limited to isolated grains



Very Low SCC Crack-Growth Rates Measured for CRDM Alloy 690 Heats



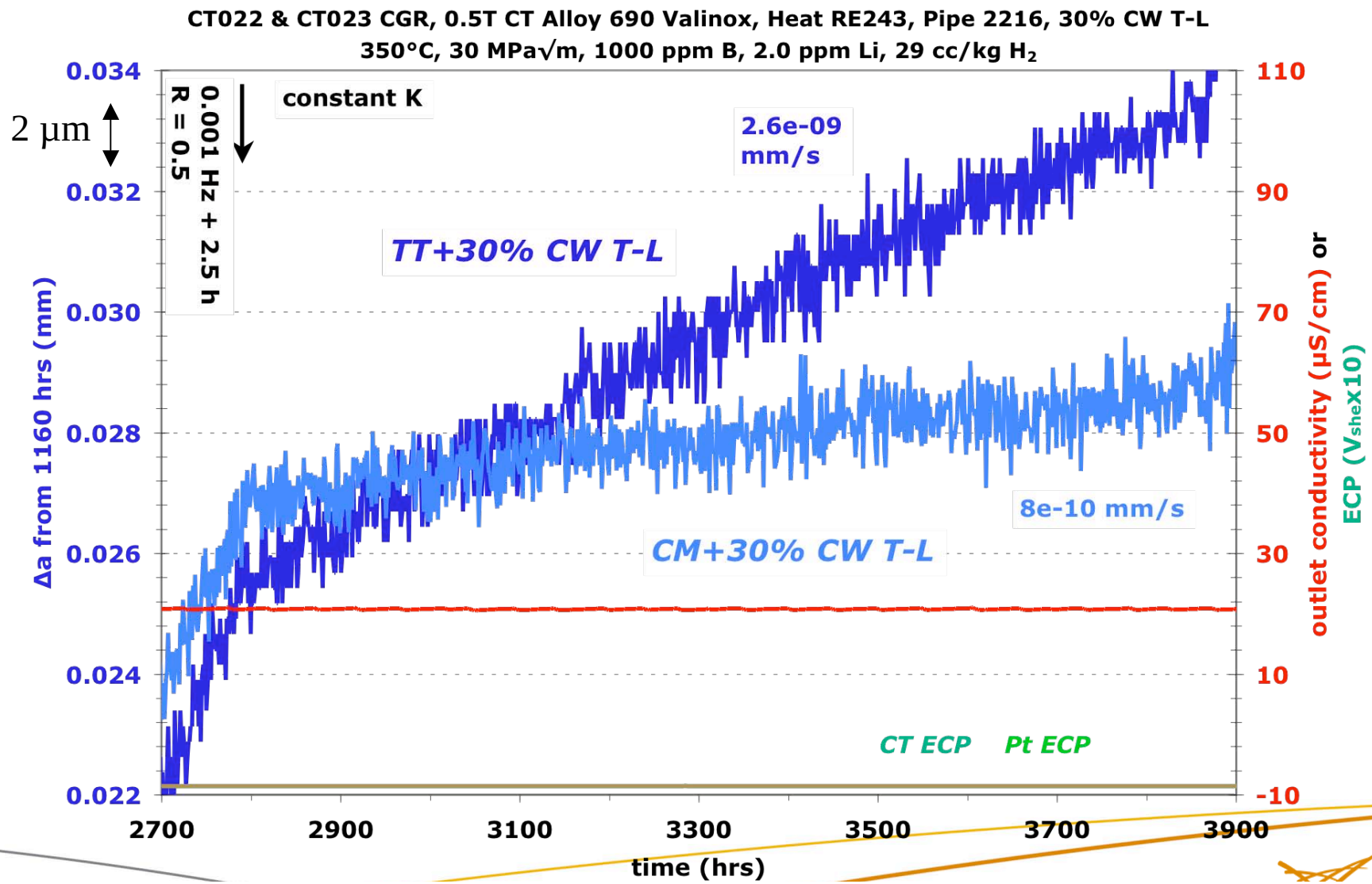
Non-CW CRDM alloy 690 heats show rates less than ~$2x10^{-9}$ mm/s (most $<5x10^{-10}$ mm/s) with only isolated regions of IGSCC

Alloy 690 Crack Growth Tests in Simulated PWR Water at PNNL

Alloy 690 Testing - 8 specimens complete, 2 on-going

- Valinox CRDM Heat RE243: specimen CT014 in a carbide-modified (CM) condition, specimen CT015 in the as-received thermally treated (TT) condition
- Valinox CRDM Heat: specimen CT026 is heat WP142 in TT condition and specimen CT027 is heat WP140 in TT condition
- **Valinox CRDM Heat RE243, specimen CT019 in TT + 17% CR (S-L) condition and specimen CT020 in CM + 17% CR (S-L) condition**
- **Valinox CRDM Heat RE243, specimen CT022 in a TT + 30% CR (T-L) condition and specimen CT023 in CM + 30% CR (T-L) condition**
- Cold-Rolled Plate: specimen CT036 - ANL heat in 26% CR (S-L) condition and specimen CT037 - GEG heat in 20% CR (S-L) condition

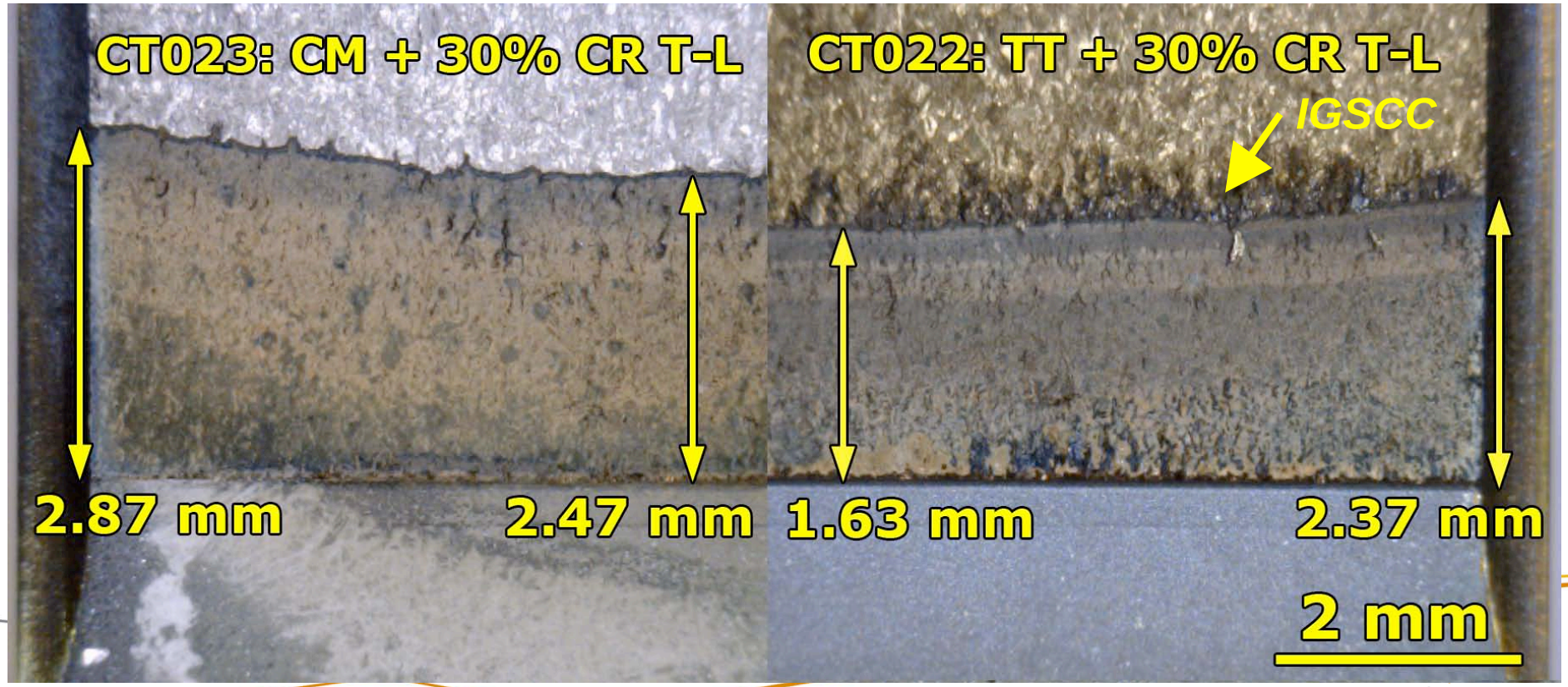
Alloy 690 Heat RE243 30% CR T-L Constant K



Higher crack-growth rate in 30% CR alloy 690TT, reproduced at higher K levels

Alloy 690 Heat RE243 30% CW T-L 350°C Crack Growth Surface

- CM + 30% CR T-L: IG cracking limited to isolated grains similar to non-CR TT and CM material.
- TT + 30% CR T-L: higher fraction of IG cracking consistent with higher observed crack growth rate.



Alloy 690 Heat RE243 30% CW T-L 350°C Crack Growth Surface

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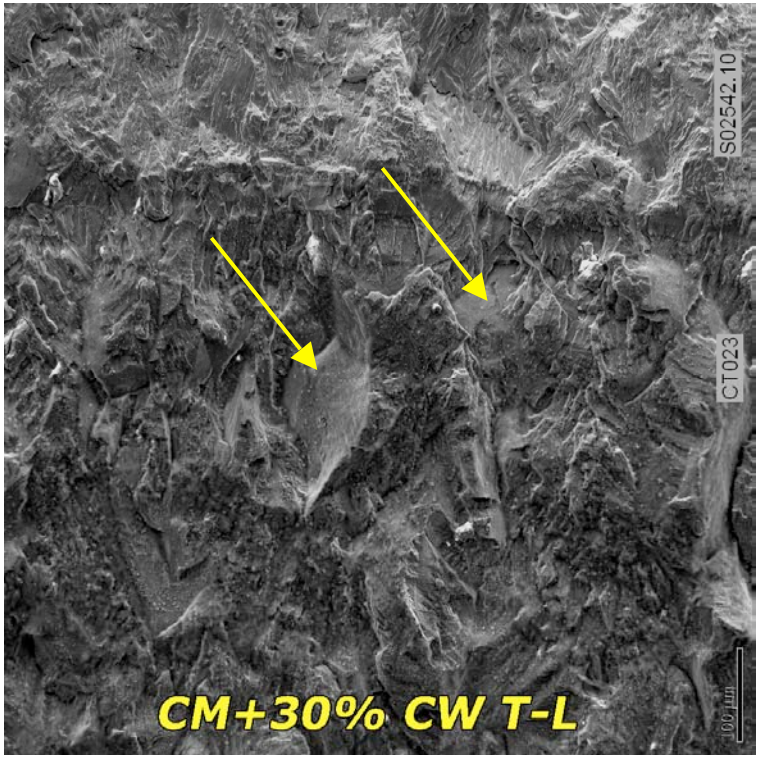
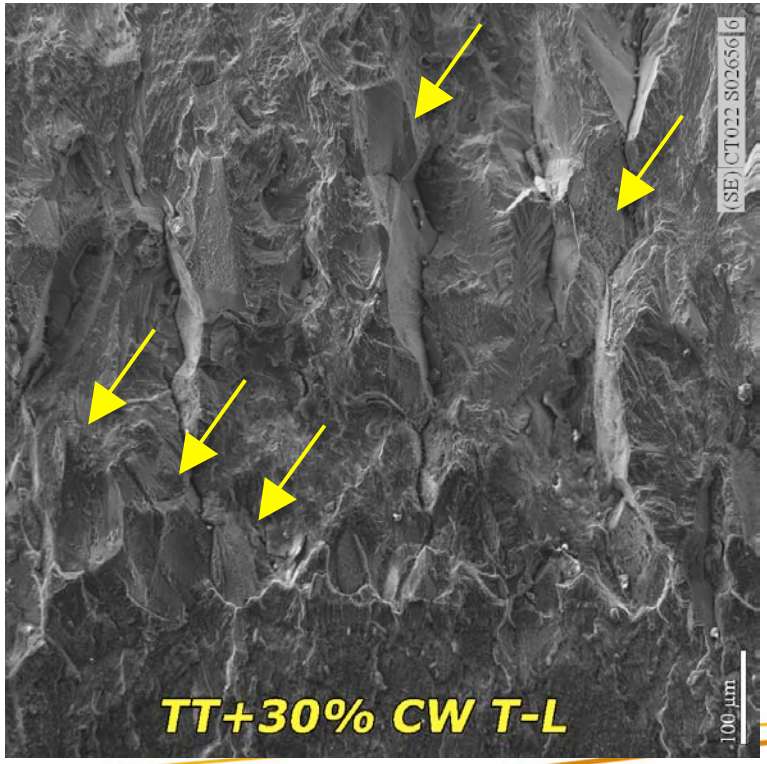
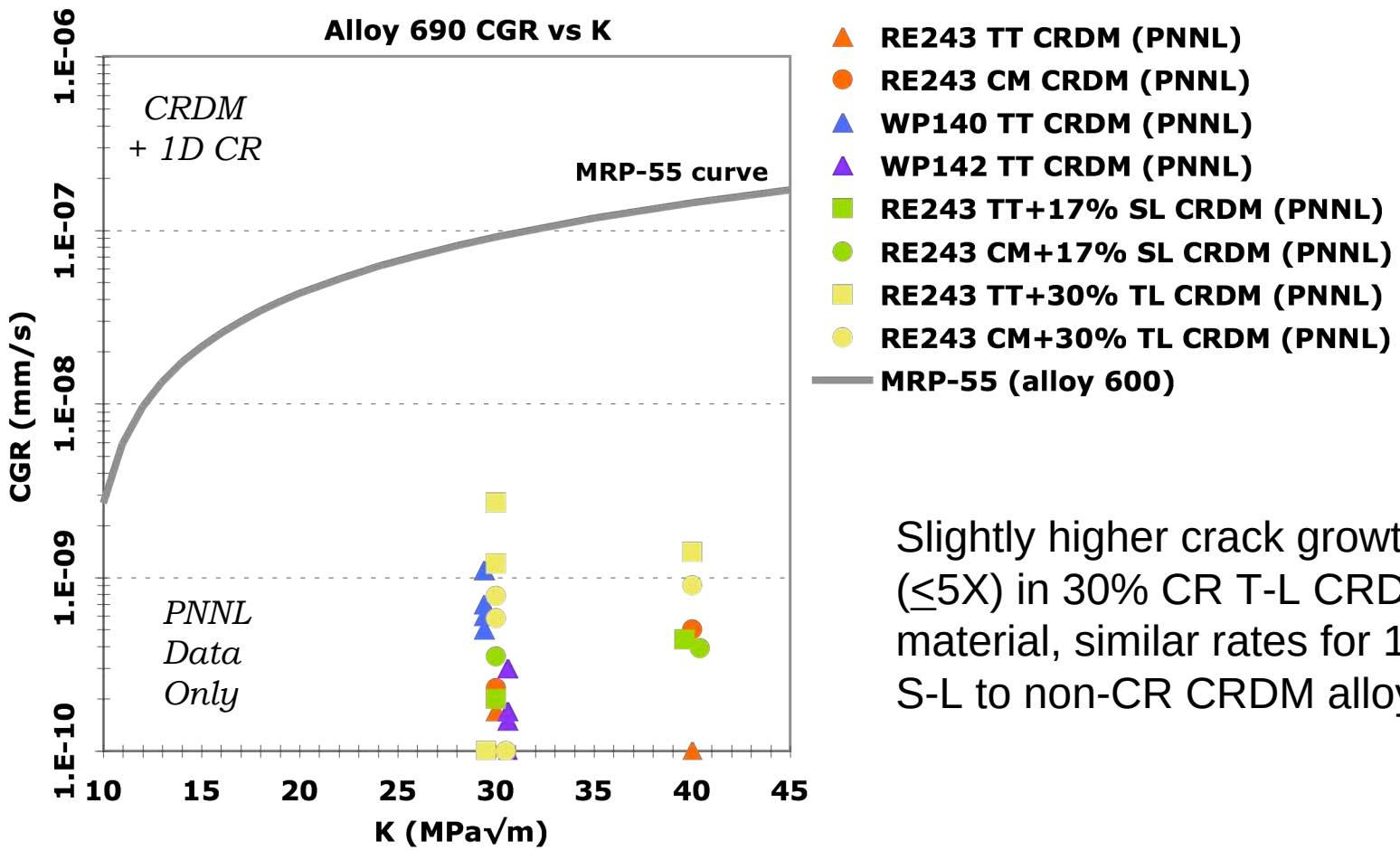




Most IG cracks on grain boundaries parallel to rolling plane.

Very Low SCC Crack-Growth Rates Measured for CRDM Alloy 690 Heats



Slightly higher crack growth rates (≤5X) in 30% CR T-L CRDM material, similar rates for 17% CR S-L to non-CR CRDM alloy 690.

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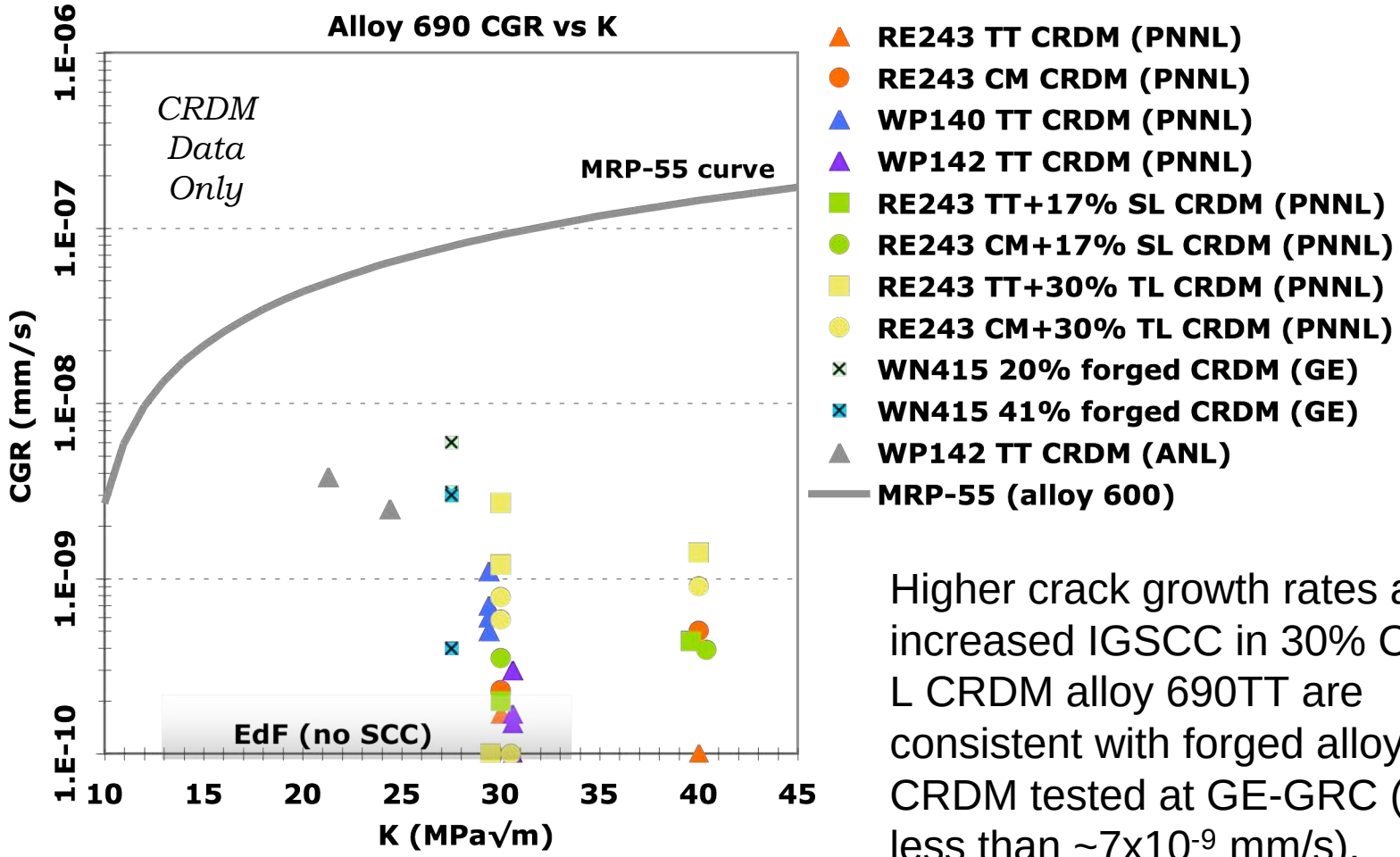
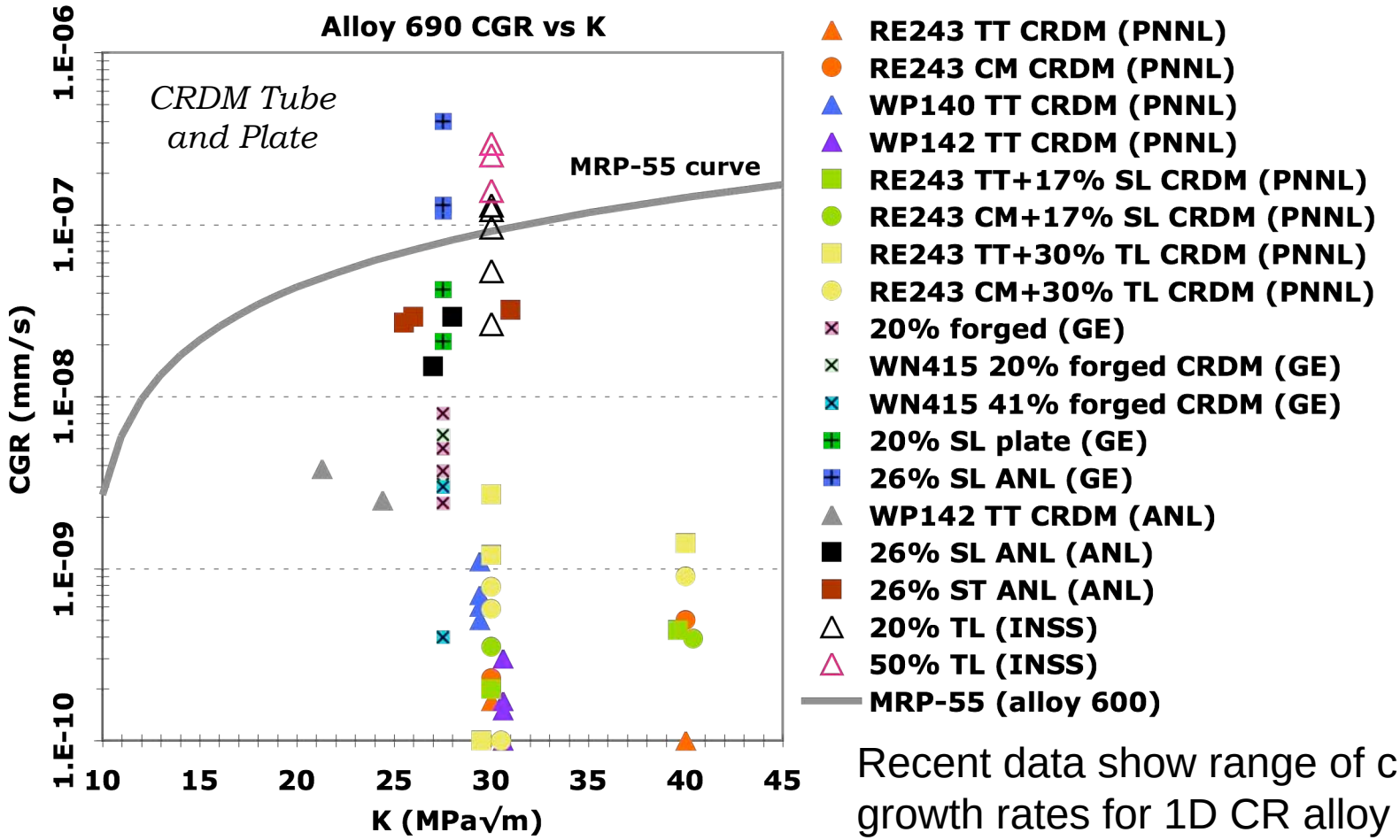
Low SCC Crack-Growth Rates Measured for CW CRDM Alloy 690 Heats



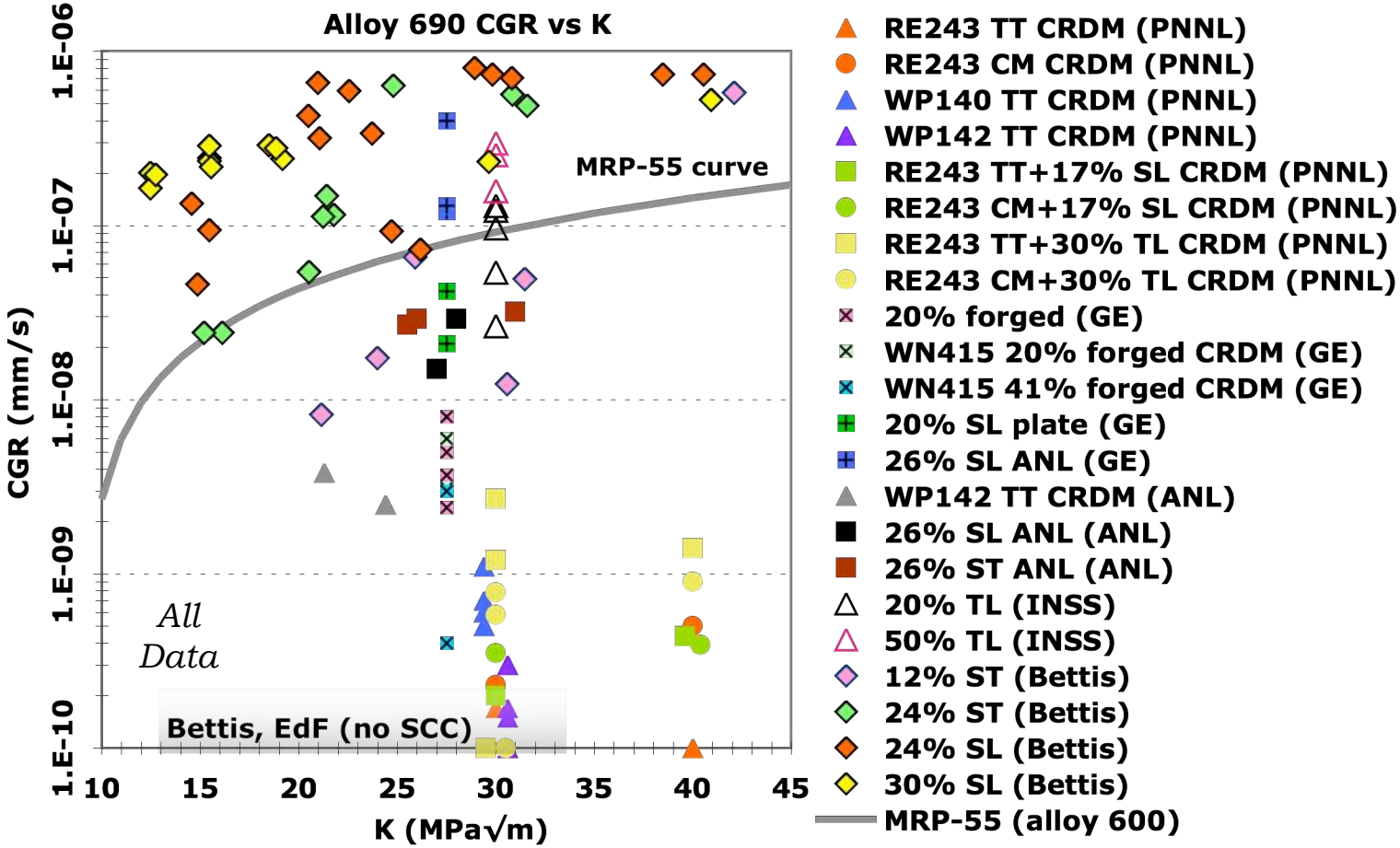
Higher crack growth rates and increased IGSCC in 30% CR T-L CRDM alloy 690TT are consistent with forged alloy 690 CRDM tested at GE-GRC (still less than ~7×10^{-9} mm/s).

High SCC Crack-Growth Rates Measured for Many 1D-CR Alloy 690 Heats



Recent data show range of crack growth rates for 1D CR alloy 690 plate, 10-500X higher than initial 1D CR CRDM results. Need more CRDM CR heats in S-L/S-T.

Extremely High SCC Crack-Growth Rates Measured for Many 1D-CR Alloy 690 Heats



High SCC susceptibility of many 1D CR alloy 690 plate heats has now been well documented, however root cause has not been determined. Therefore, implications for alloys in LWR service remain uncertain.

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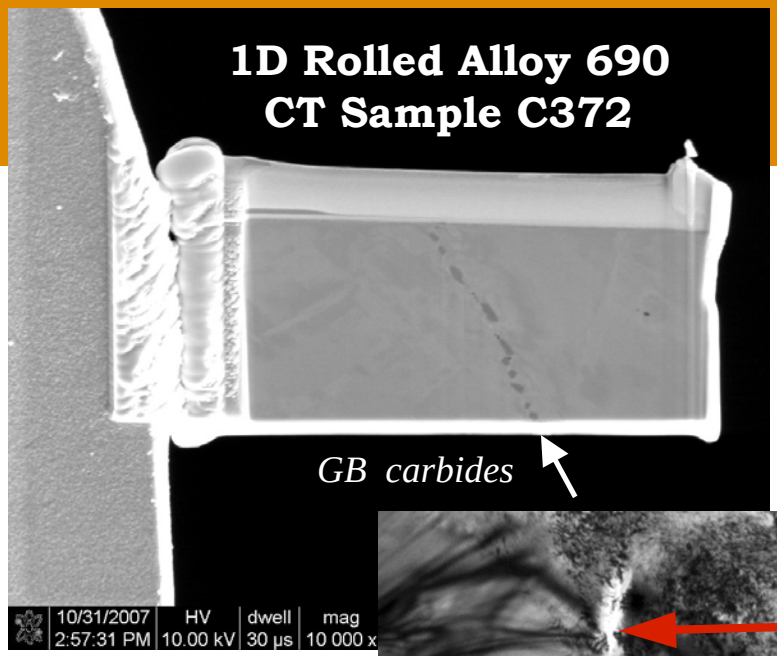
Alloy 690 SCC Crack-Growth Summary

- Both as-received TT and carbide-modified CRDM materials were evaluated in non cold-rolled and cold-rolled conditions.
- Extremely slow but generally stable propagation rates in alloy 690 CRDM materials (three different heats).
  - Non cold-rolled materials have constant K CGRs of $<1 \times 10^{-9}$ mm/s.
  - Carbide modification treatment does not increase crack growth rate.
  - 17% CR S-L has same crack growth rate as non cold-rolled material.
  - TT + 30% CR T-L showed ~5X higher growth rates ($\sim 3 \times 10^{-9}$ mm/s), may have been significantly higher if tested in S-L orientation.
- Transgranular cracking dominates in as-received alloy 690 CRDM heats with only isolated IG regions. More extensive IG cracking was observed in 30% CR T-L alloy 690.
- Limited results on the CR CRDM materials reveal much lower crack-growth rates (10-500X) than for 1D CR alloy 690 plate.

Alloy 690 Characterization Activities

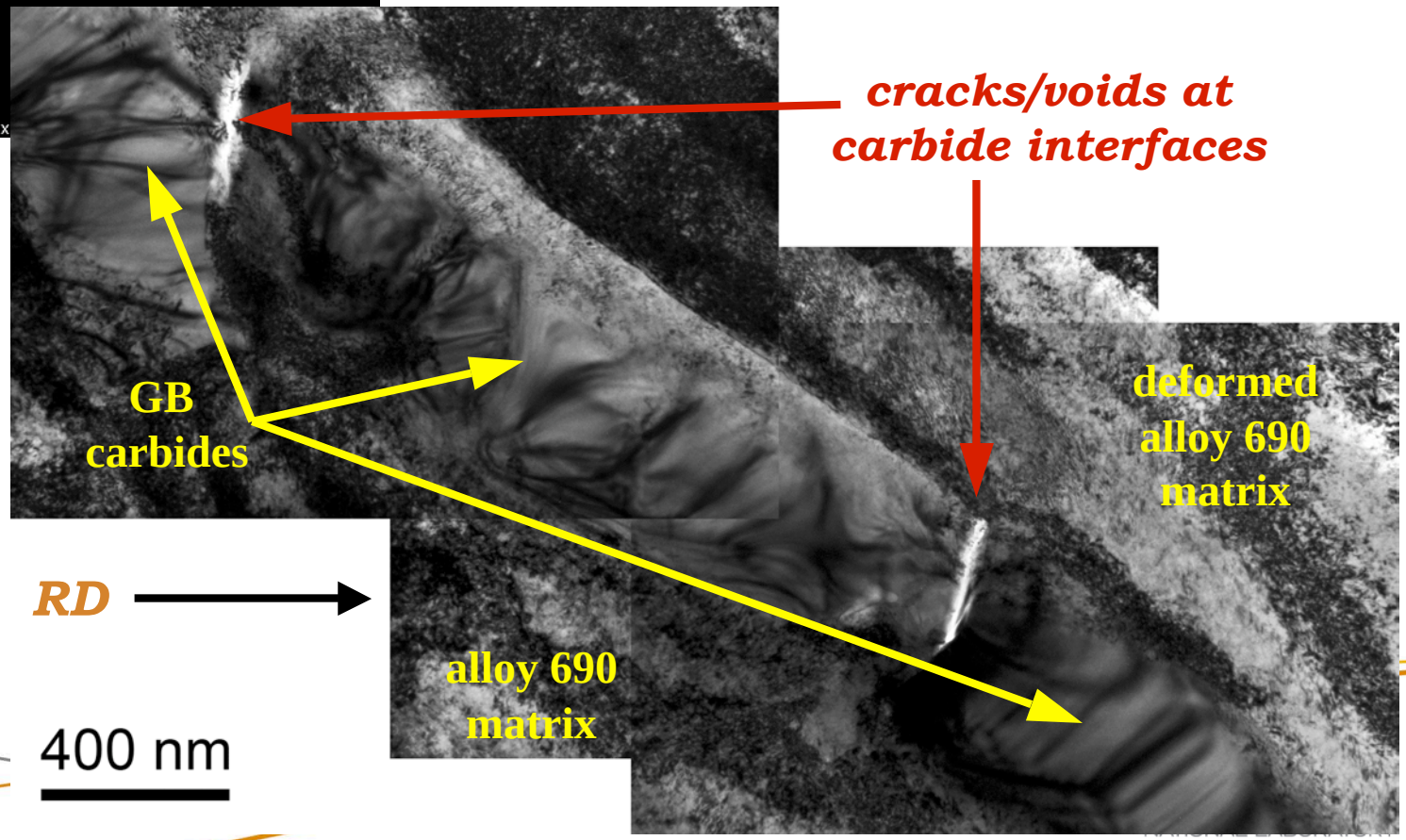
- Optical metallography, SEM, EBSD and ATEM
- Alloy 690 CRDM Heats
  - As-received TT condition has similar grain size and GB carbides.
  - Carbide modification treatment slightly reduces GB carbide density.
  - Limited microstructure and hardness change after 17% CR, noticeable after 30%.
- Alloy 690 Cold Rolled Plates
  - 26% 1D CR ANL heat shows grain elongation in rolling plane, high matrix and GB strains, extensive cracking of matrix Ti-nitride particles, >~1 μm spaced cracks/voids at semi-continuous IG carbides.
  - 20% 1D CR GEG heat shows high matrix and GB strains, limited Ti-nitride particles or IG carbides, only isolated GB cracks/voids.
  - Rolling-induced damage can create a pre-cracked microstructure, however far from a continuous path and mechanism remains unclear to explain the ~100+X increase in SCC propagation rates.

Grain Boundary Damage in 1D CR Alloy 690 Heats

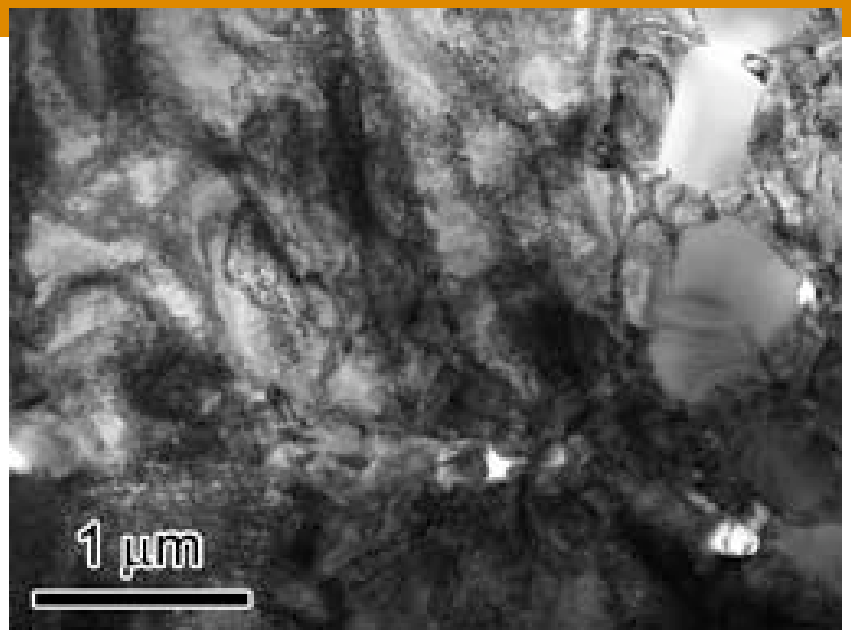


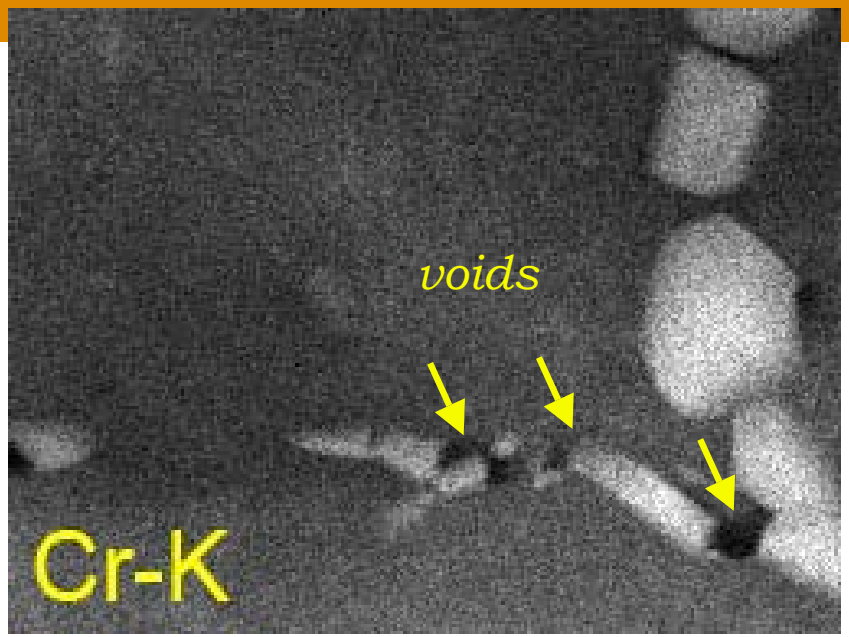
Focused ion beam (FIB) and TEM has been used to evaluate nm-scale damage along grain boundaries in 1D-rolled alloy 690 that show high IGSCC crack-growth rates during tests in PWR primary water. ***Nanoscale cracks and/or voids*** *found by TEM at grain boundary carbide interfaces.*

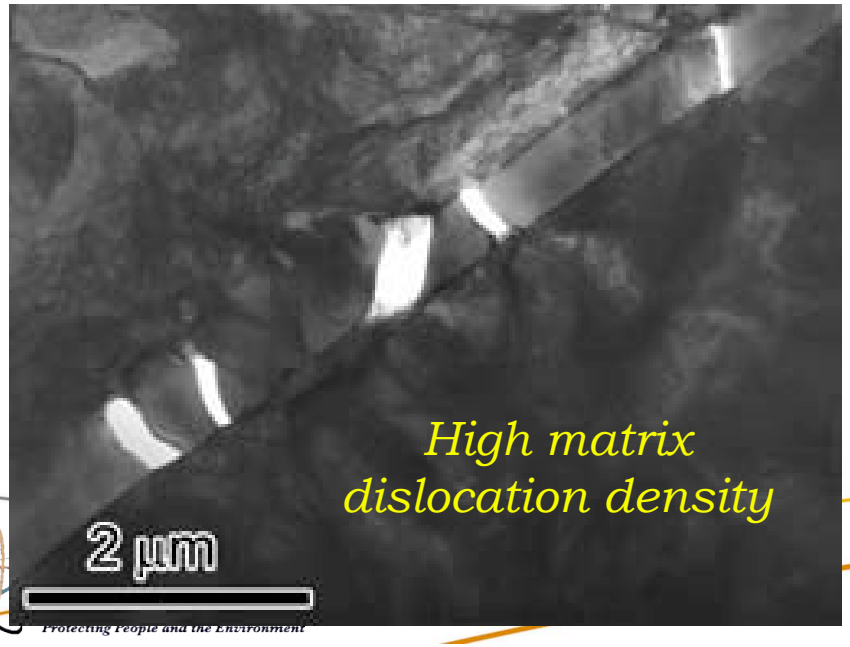
FIB has removed samples from regions far from the CT fracture surface. Samples cut from several different section orientations to assess the 3D grain boundary characteristics.

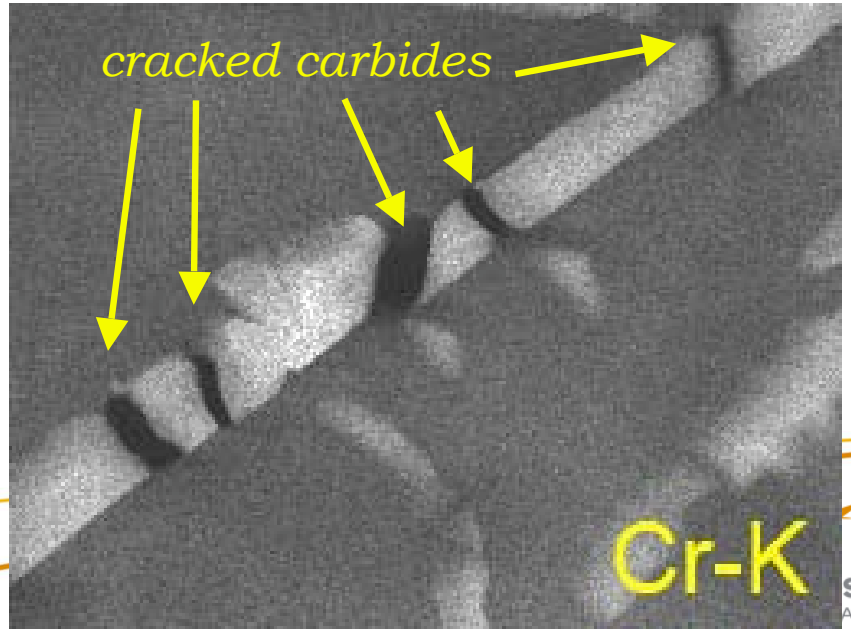


Alloy 690 Plate 26% Cold Rolled

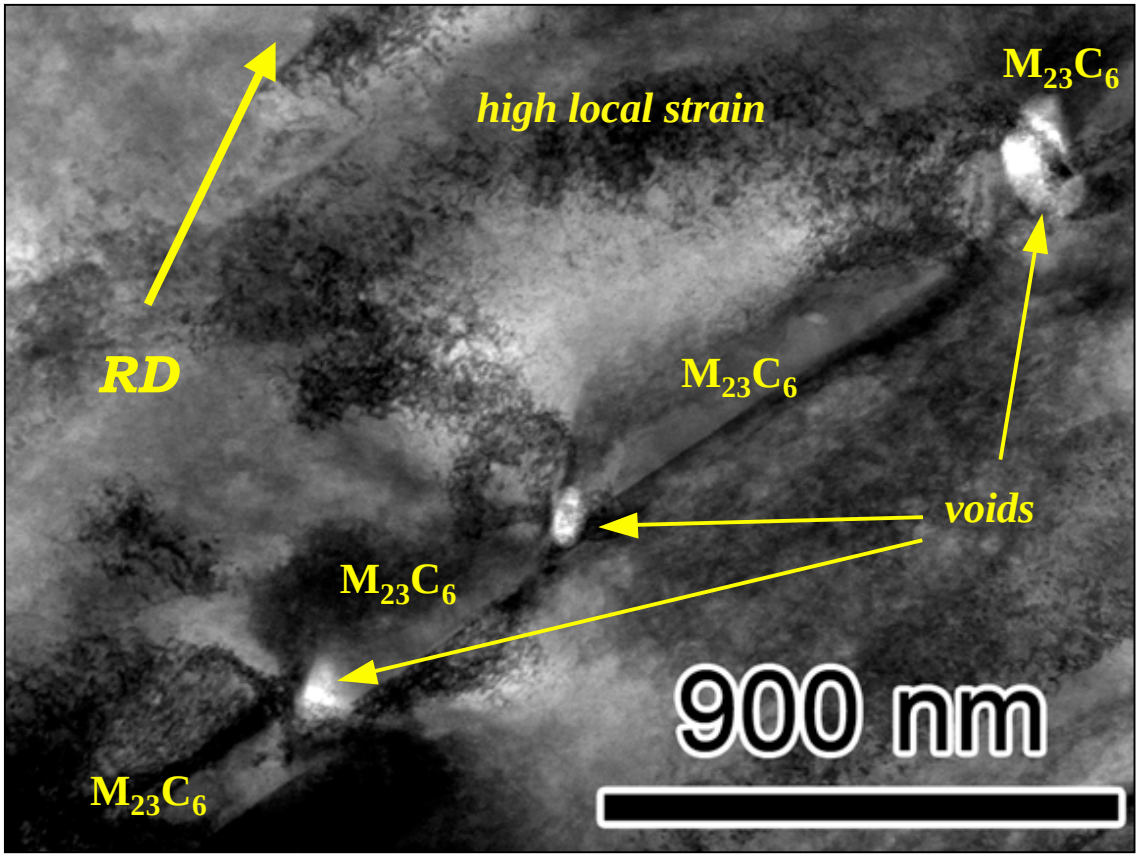








Alloy 690 Plate 26% Cold Rolled



TEM image from third series of FIB samples (two orientations); voids separating carbides, high local strain at voids and at carbide interfaces.

Alloy 690 Characterization Activities

- Optical metallography, SEM, EBSD and ATEM
- Alloy 690 CRDM Heats
  - As-received TT condition has similar grain size and GB carbides.
  - Carbide modification treatment slightly reduces GB carbide density.
  - Limited microstructure and hardness change after 17% CR, noticeable after 30%.
- Alloy 690 Cold Rolled Plates
  - 26% 1D CR ANL heat shows grain elongation in rolling plane, high matrix and GB strains, extensive cracking of matrix Ti-nitride particles, >~1 μm spaced cracks/voids at semi-continuous IG carbides.
  - 20% 1D CR GEG heat shows high matrix and GB strains, limited Ti-nitride particles or IG carbides, only isolated GB cracks/voids.
  - Rolling-induced damage can create a pre-cracked microstructure, however far from a continuous path and mechanism remains unclear to explain the ~100+X increase in SCC propagation rates.

PNNL Alloy 690 Plans 2009-2010

- Evaluate effects of 1D cold rolling, tensile straining and forging on SCC in alloy 690 CRDM and plate heats.
  - Complete tests on ANL and GEG 1D CR plate for crack-tip exams.
  - New tests on 1D 30%CR CRDM heat 243 plus 2nd CRDM heat.
  - New tests on tensile-strained plate materials (EPRI or from collaborative program), compare response to 1D CR and to forged.
- Evaluate alloy 690 HAZ SCC response (KAPL narrow gap weld) and correlate with respect to deformation effects.
- Continue low-to-high resolution characterizations linking matrix and grain boundary microstructure-microchemistry to SCC response and help determine root cause for high susceptibility including crack-tip examinations.

Alloy 152/52/52M Materials

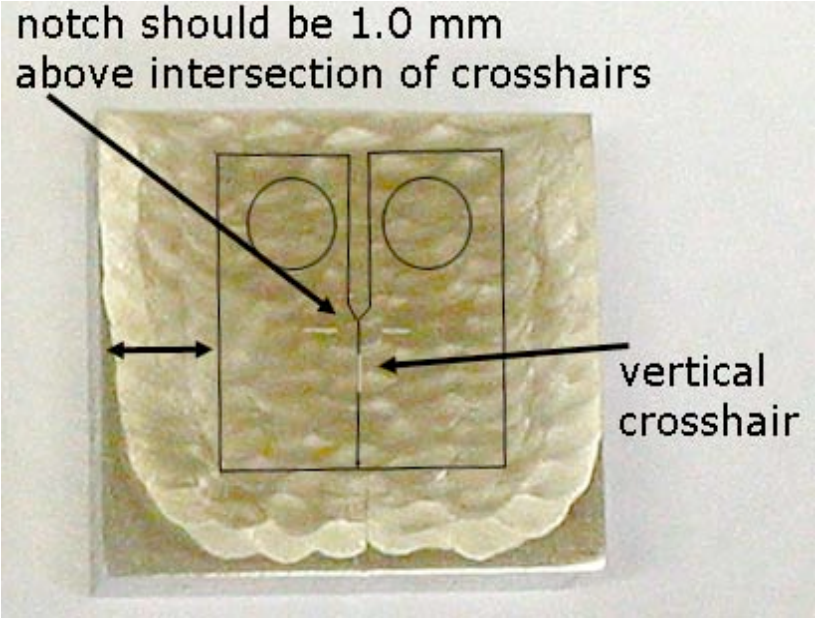
- Weld metal sources (industry mockups)
  - Alloy 152 and alloy 52 U-groove mockups made by MHI for Kewaunee
  - Alloy 52 butt weld made by AREVA
  - Alloy 52M overlay and inlay mockups made by Ringhals
- Compositions
  - MHI Alloy 152: 9.1Fe, 28.7Cr, 0.03C, 3.6Mn, 0.33Si, 0.15Al, 0.12Ti, Bal Ni
  - MHI Alloy 52: TBD
  - AREVA Alloy 52: 10.0Fe, 29.1Cr, 0.024C, 0.25Mn, 0.13Si, 0.71Al, 0.52Ti, Bal Ni
  - Ringhals Alloy 52M/182 Overlay, Alloy 52M: 8.42Fe, 30.0Cr, 0.020C, 0.81Mn, 0.03Si, 0.10Al, 0.21Ti, 0.85Nb, 0.15Zr, Bal Ni
  - Ringhals Alloy 52M/82 Inlay: TBD
- Thermomechanical conditions
  - All specimens but one tested in as-welded condition
  - One alloy 152 specimen heat treated at 610°C/12 h (LAS stress relief)

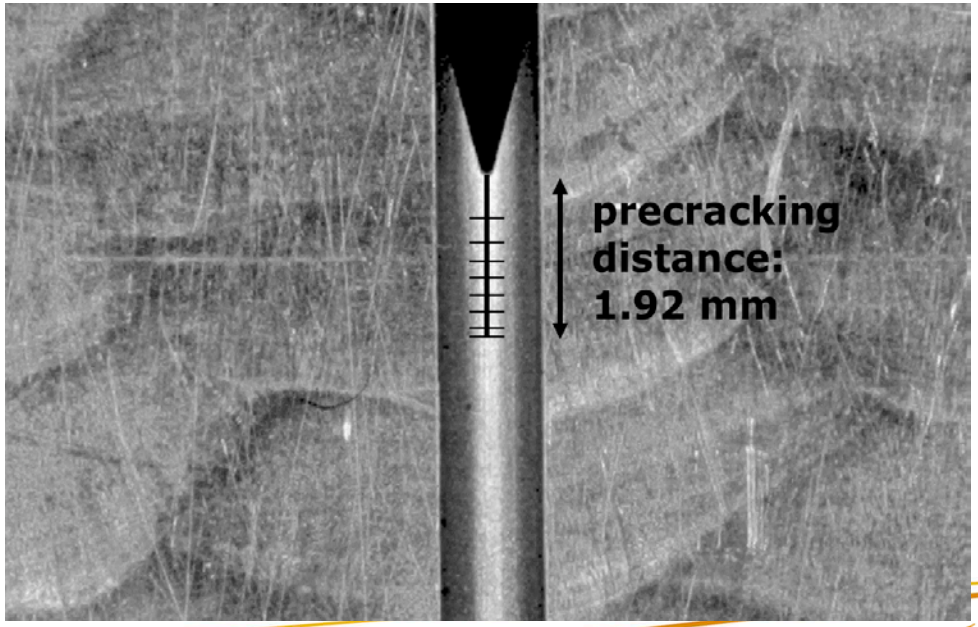
Alloy 152/52/52M Crack Growth Tests in Simulated PWR Primary Water at PNNL

- **Alloy 152/52/52M - 7 specimens complete, 6 on-going**
  - MHI alloy 152 mockup: specimen CT013 in as-welded (AW) condition
  - MHI alloy 152 mockup: specimen CT017 in AW + LAS heat treatment (610°C/12 h/AC) condition, specimen CT018 in AW condition
  - Alloy 52: specimen CT024 is AREVA mockup in AW condition, specimen CT025 is MHI mockup in AW condition
  - Alloy 52M from Ringhals alloy 182/52M overlay mockup: CT028 & CT029 both in AW condition
  - Alloy 52M from Ringhals alloy 82/52M inlay mockup: CT030 & CT031 both in AW condition (on going in alloy 52M)
  - Alloy 52: specimen CT032 is AREVA mockup in AW condition, specimen CT033 is MHI mockup in AW condition (on going)
  - Ringhals alloy 182/52M overlay mockup: CT034 & CT035 both in AW condition (on going in alloy 182 approaching alloy 52M interface)

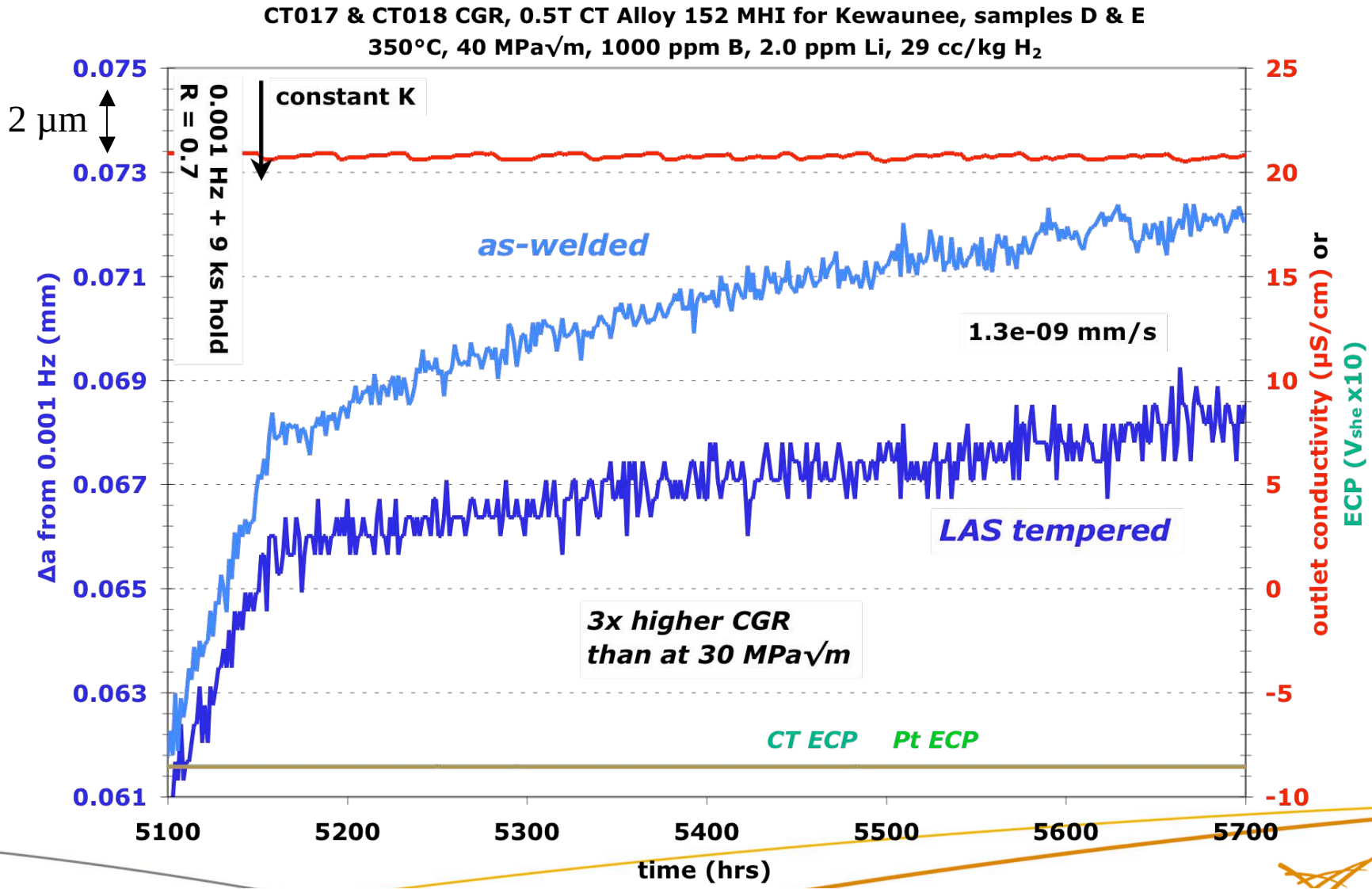
Alloy 152/52 Weld Specimens

- Notch and precrack positioned to allow SCC growth to begin in the middle of a weld pass.
- Crack oriented along dendrite direction as best as possible.
- In some instances, specimens were machined to evaluate different weld passes.



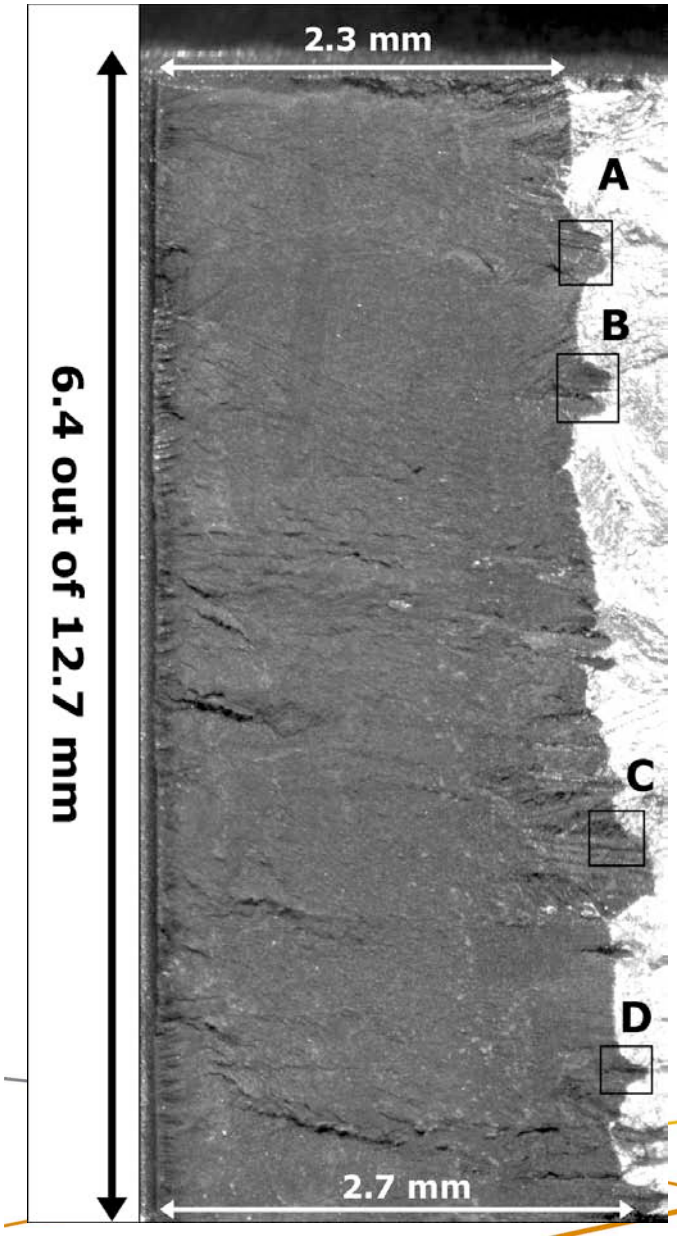


MHI Alloy 152 Test #2
Constant K at 350°C, 40 MPa√m

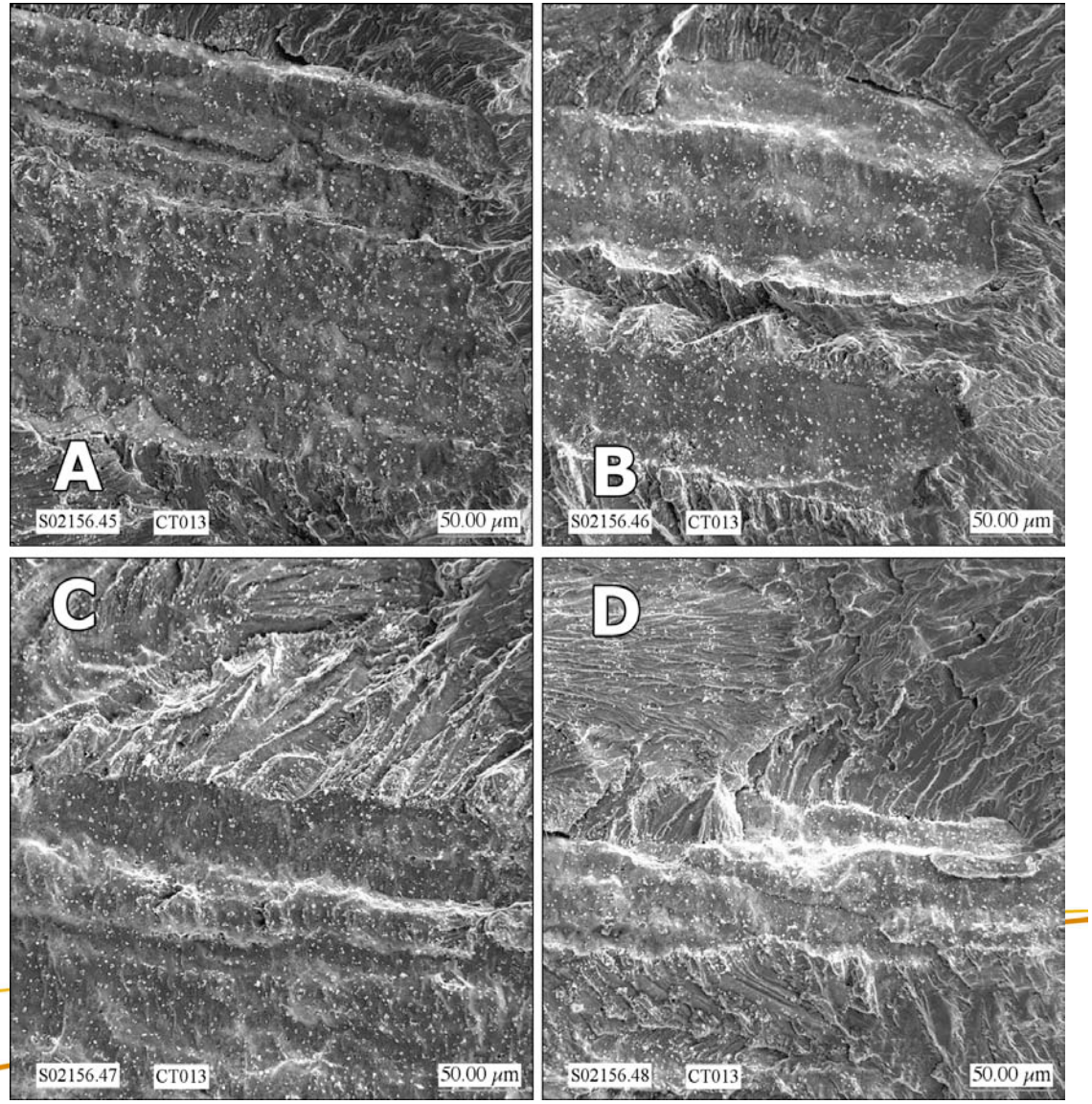


Higher SCC growth rates at higher K, remains low (~1x10^{-9} mm/s)

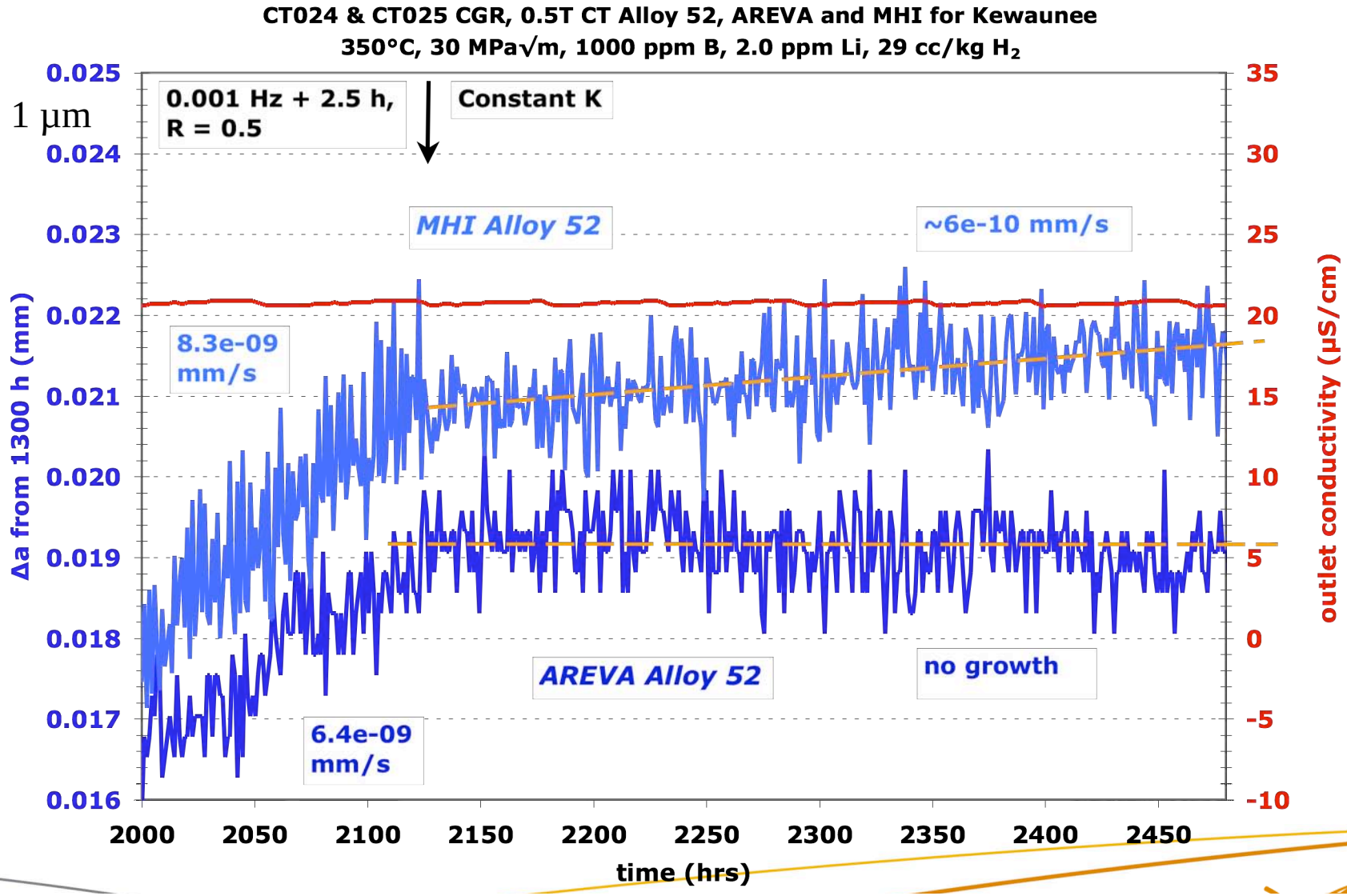
MHI Alloy 152 Test #1
350°C Crack Growth Surface



Approximately 35% IG engagement

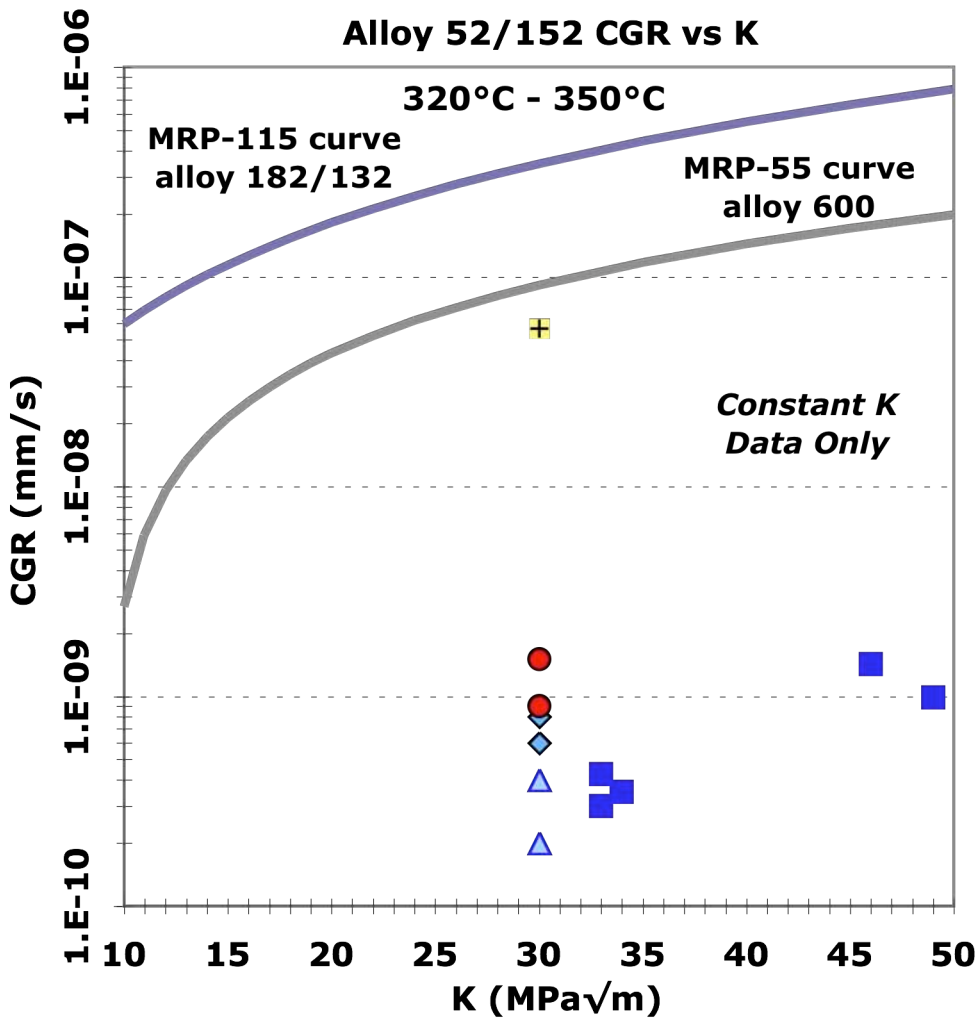


AREVA Alloy 52 and MHI Alloy 52



Consistently different response for two welds, but very low crack growth rates

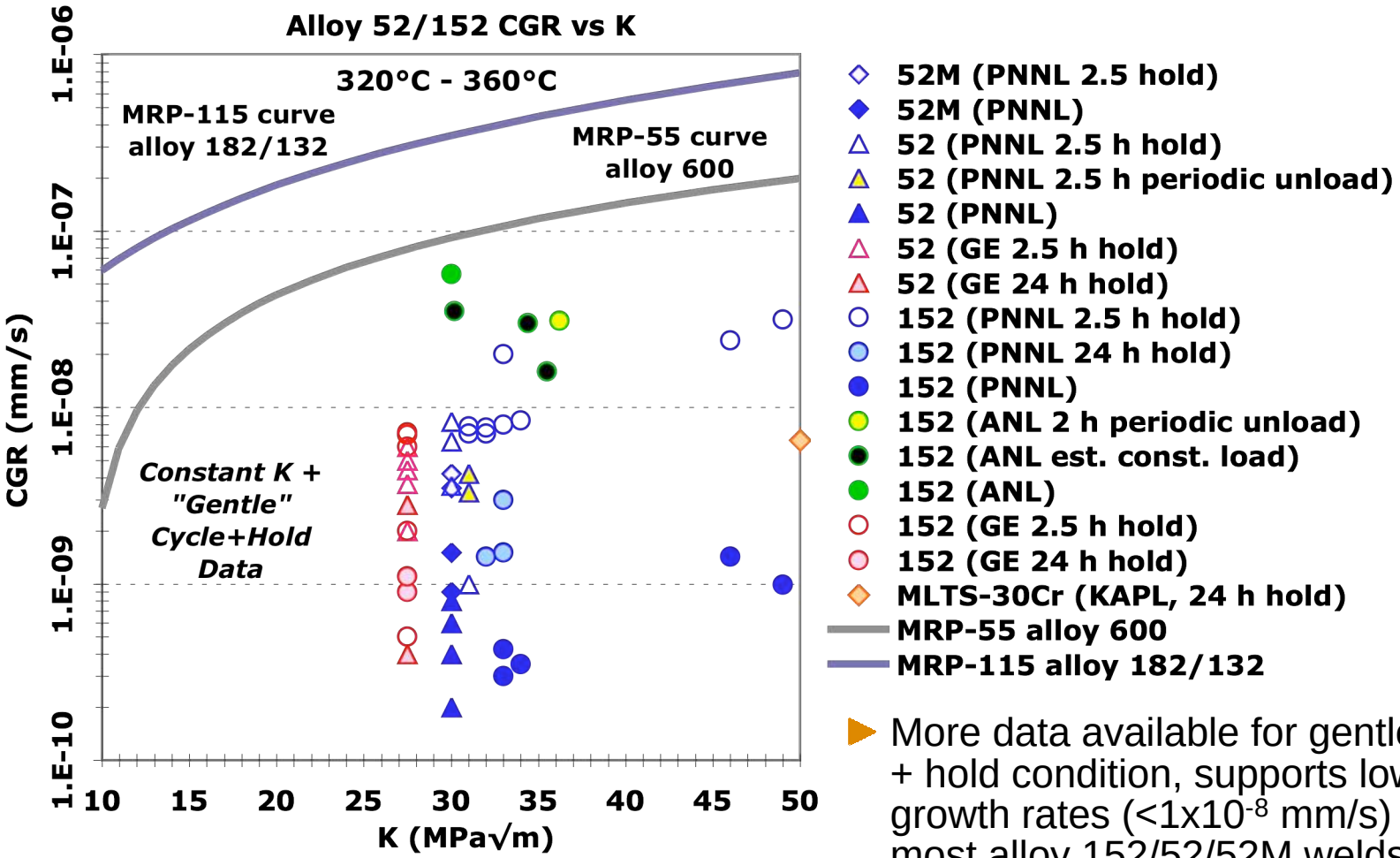
Alloy 152/52/52M Crack Growth Rate vs K Constant K or Constant Load Only



- alloy 152 MHI (PNNL)
- alloy 52 MHI (PNNL)
- alloy 52 AREVA (PNNL)
- alloy 52M Ringhals inlay (PNNL)
- alloy 152 (ANL)
- MRP-55 alloy 600
- MRP-115 alloy 182/132

- Possible K dependence for 152, but rates at higher K are still very low.
- Fractography indicates engaged (IG) CGR for alloy 152 is ~3x the ***average*** CGR. The ***maximum*** CGR is up to 6x the ***average*** CGR. Even the "conservative maximum" rates are <~$1x10^{-8}$ mm/s.
- Constant K SCC data on alloy 152/52/52M remains very limited.

Alloy 152/52/52M Crack Growth Rate vs K Expanded Data



- More data available for gentle cycle + hold condition, supports low crack growth rates (<1x10^{-8} mm/s) for most alloy 152/52/52M welds.
- Higher growth rates seen for certain alloy 152 welds, more data needed.

Alloy 152/52/52M Summary

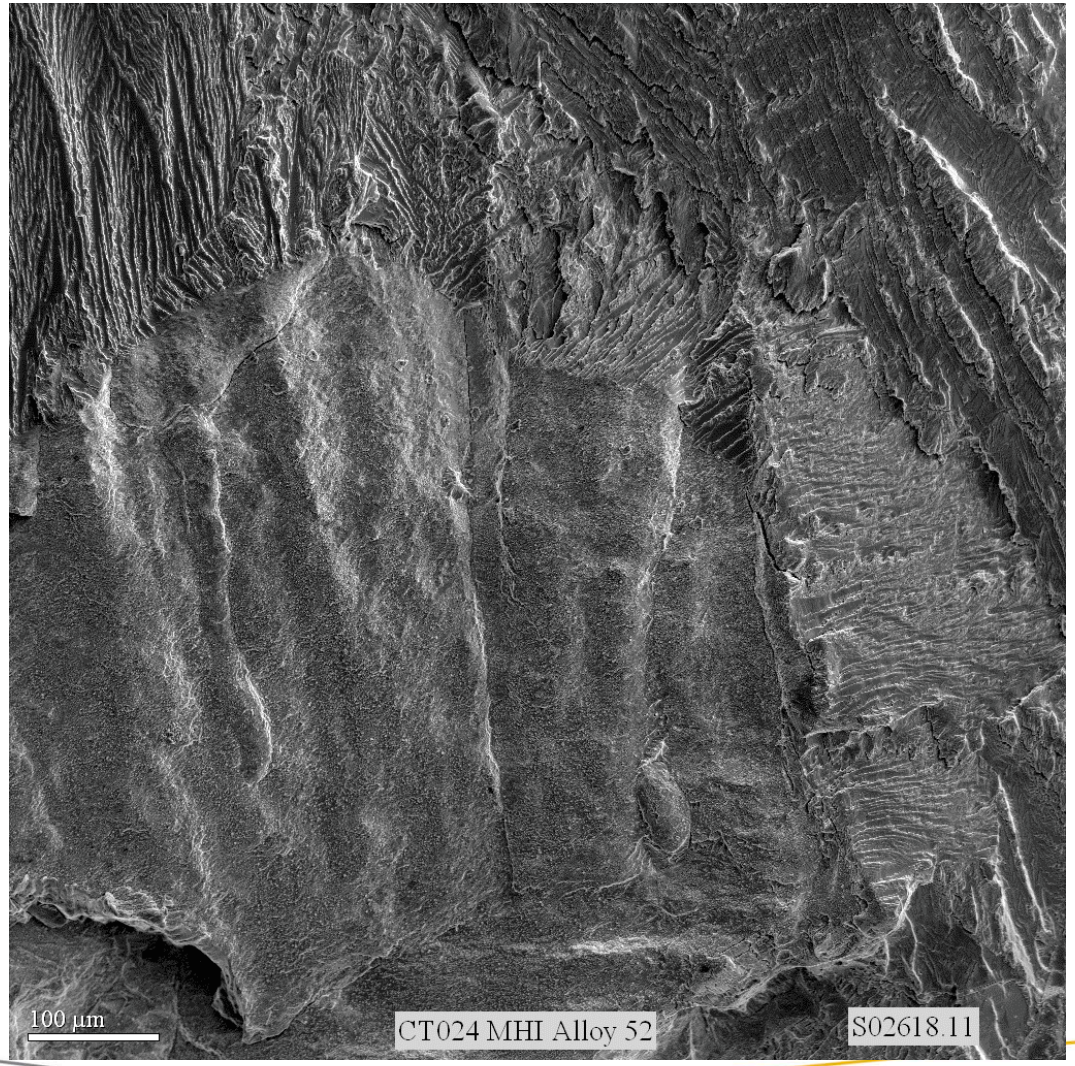
- Very slow SCC propagation in weld metals for industry mockups in simulated primary PWR primary water even when the crack is oriented along dendrite boundaries in a single pass.

- Stable IG crack advance observed at 350°C under gentle cycle + hold and constant K conditions, but at very low growth rates.
  - Highest constant K rates (~2×10^{-9} mm/s) for MHI alloy 152 and inlay alloy 52M (based only on dcpd).
  - Alloy 52 welds and alloy 52M overlay show lower rates.
  - Some weld-to-weld differences, no influence of LAS stress relief.

- IG cracking produced during cycling at 50°C.
  - Alloy 152 most susceptible, different behavior for alloy 52 welds.
  - Significant IG engagement at 50°C did not promote more IGSCC at 350°C.

MHI Alloy 52 Crack Growth Surface

IG Cracking during 50°C Cycling, Limited IGSCC Growth at 350°C



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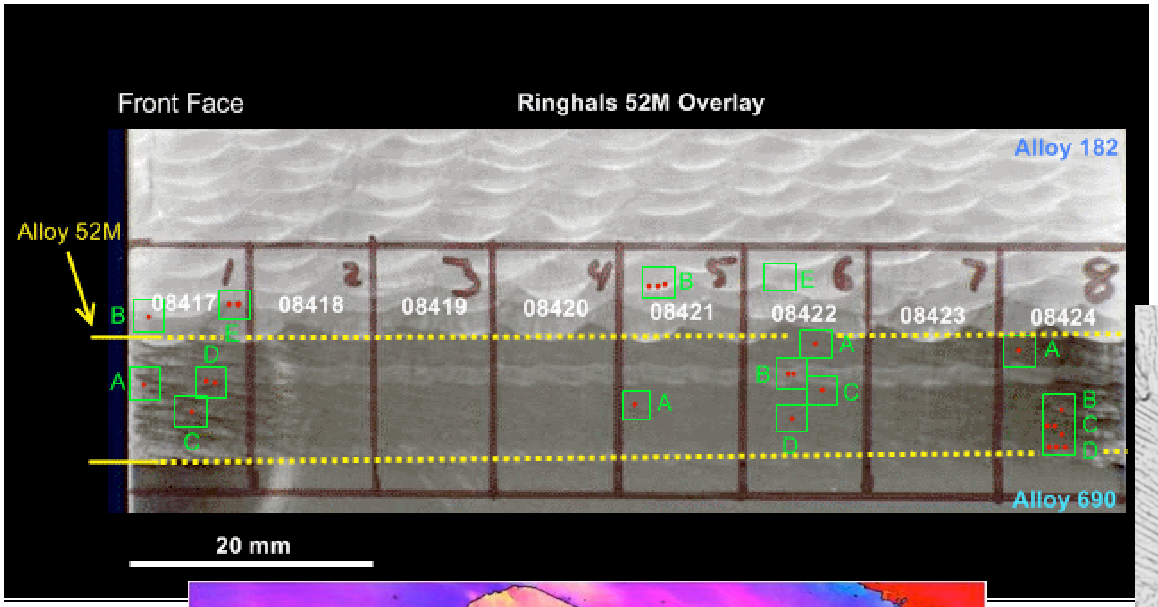
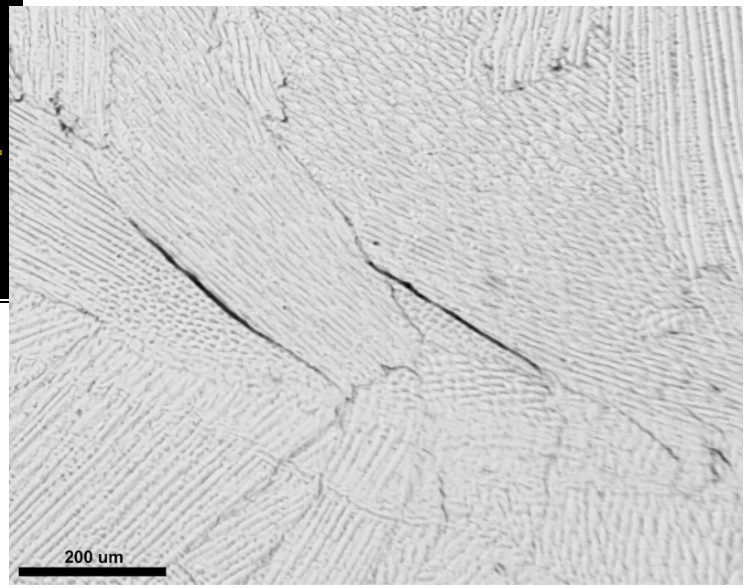
Weld Metal Characterization Activities

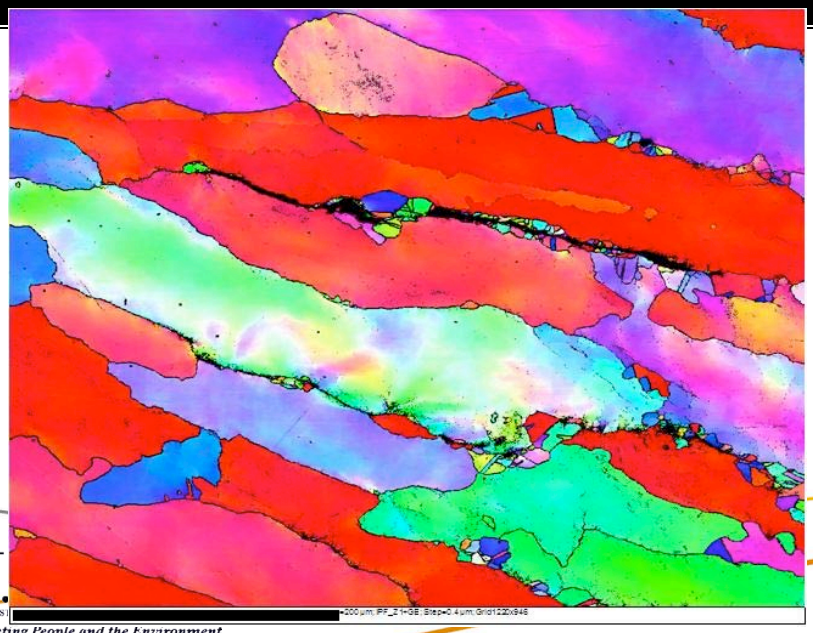
- Optical metallography, SEM, EBSD and TEM.
- Alloy 152 and 52 Weld Mockups
  - IG weld cracks in alloy 52 weld associated with fit-up region for SS plate, no cracks in alloy 152 weld or alloy 52 butt weld.
  - Limited solidification segregation, strong variation is GB carbides
  - Complex microstructures in dilution zones with 304SS
- Alloy 182/52M Overlay and Alloy 82/52M Inlay Mockups
  - IG weld cracks in localized regions for alloy 52M
  - Limited solidification segregation, strong variation is GB carbides
  - Narrow dilution zones (~100 μm) with alloy 182 or 82.
- 304SS/Alloy 52 Overlay
  - Extensive dilution zone (~1 mm) between alloy 52 and stainless steel
  - IG weld cracks in dilution zone associated with elongated TiN at GBs

Weld Metal Defect Characterizations



Optical metallography of serial sections used to indicate distribution and morphology of weld defects

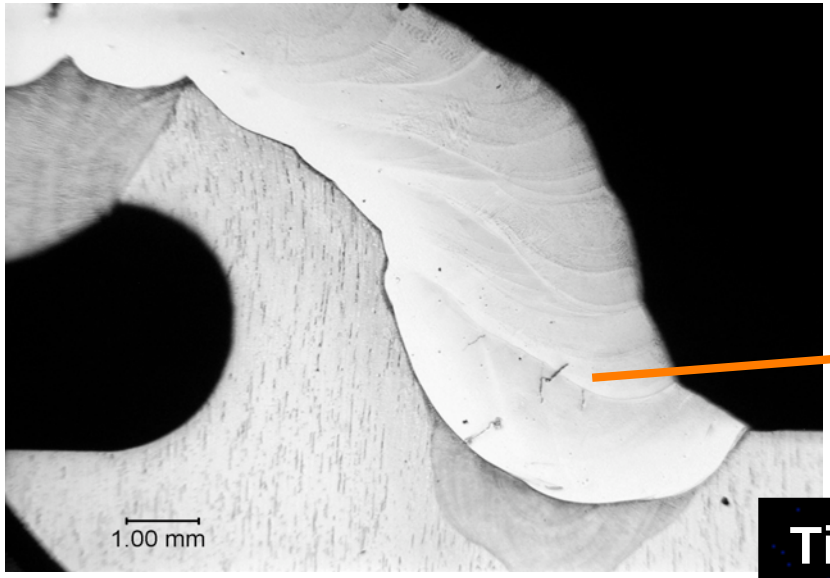


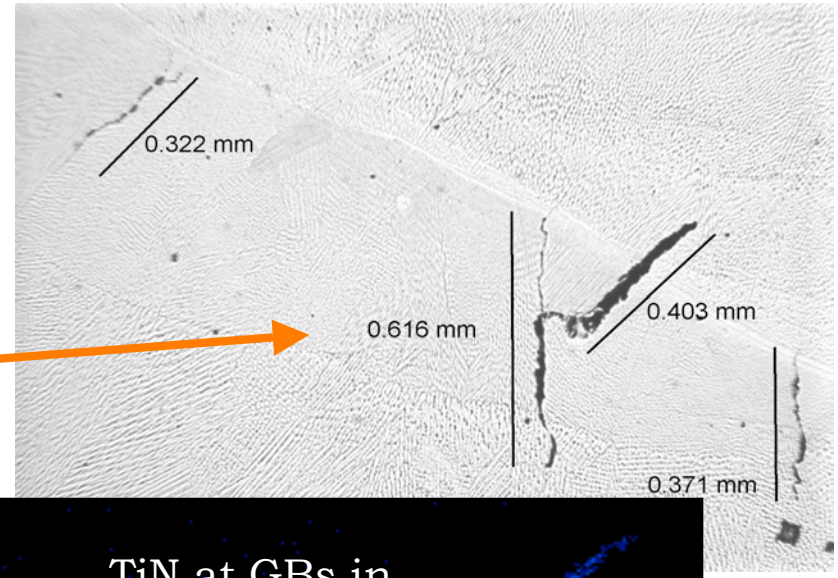


More detailed examinations by SEM, EBSD and TEM.

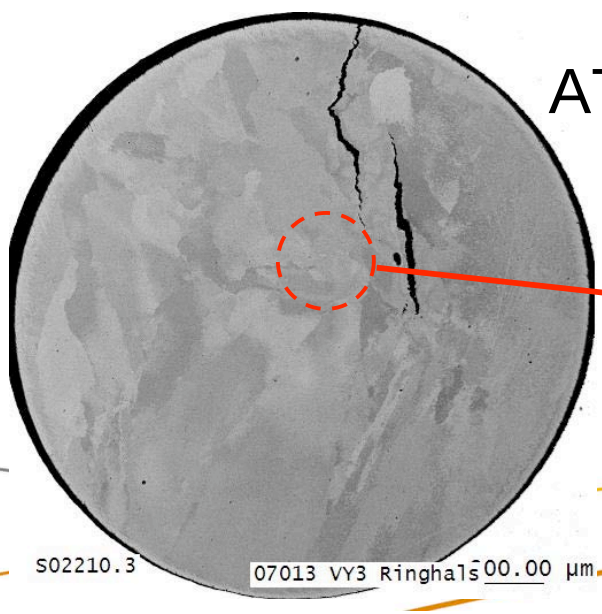
Weld Overlay Repair Mockup using Alloy 52 on Stainless Steel: Hot Cracks in Dilution Zone

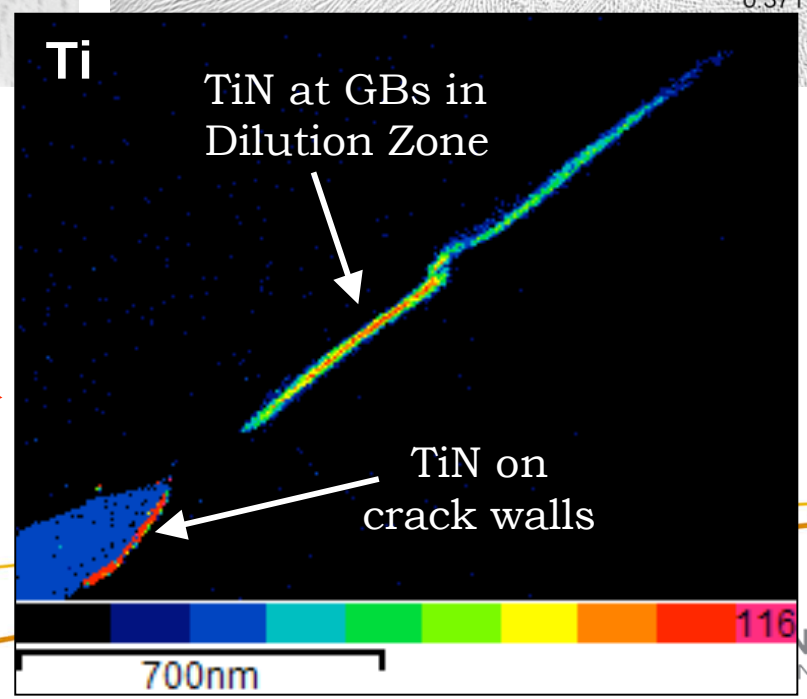
obtained from Pål Efsing and Björn Forssgren, Ringhals AB





ATEM





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PNNL Alloy 152/52/52M Plans 2009-2010

- Expand evaluation of SCC response in weld metals
  - Complete tests on Ringhals overlay and inlay mockups, establish alloy 52M response and propagation through dilution zone.
  - New tests on more susceptible alloy 152 weld metal (PNNL/ANL)
  - New tests on alloy 152/52/52M welds (e.g., from KAPL) and on new mockups welds, overlays and inlays as possible.
- Examine hot cracks and dilution zones in new KAPL welds and evaluate effects of weld defects on SCC crack growth.
- Continue low-to-high resolution characterizations linking matrix and grain boundary microstructure/microchemistry to SCC response and help determine root cause for high SCC susceptibility including crack-tip examinations.